Index:

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Mr Ades works as a Senior Marketing Manager for Wastewater & Sludge Municipal in Kemira Industry & Water segment. He has 20+ years of experience in the water, wastewater and sludge treatment field. All of his career he has worked on different positions in Kemira with focus on production, sales, product management and now marketing. He has worked in France and since 8 years with a responsibility for EMEA region.
He is representing Kemira in the Strategic Communication Committee of INCOPA, the INorganic COagulant Producer Association, based in Brussels, and part of CEFIC, the European Chemical Industry Council.

Use of iron and aluminium coagulants in wastewater treatment
Phosphorus removal and challenges of phosphorus recovery

Jean-Christophe Ades[1]
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Iron and aluminium coagulants are used in wastewater treatment since decades and they have played a key role in avoiding waterways eutrophication. Inorganic coagulants are needed to achieve ultra-low levels of phosphorus discharge, they also guarantee high phosphorus recovery rate (>50%). Finally, they allow higher energy recovery from organic waste and hence lead to a lower carbon footprint.

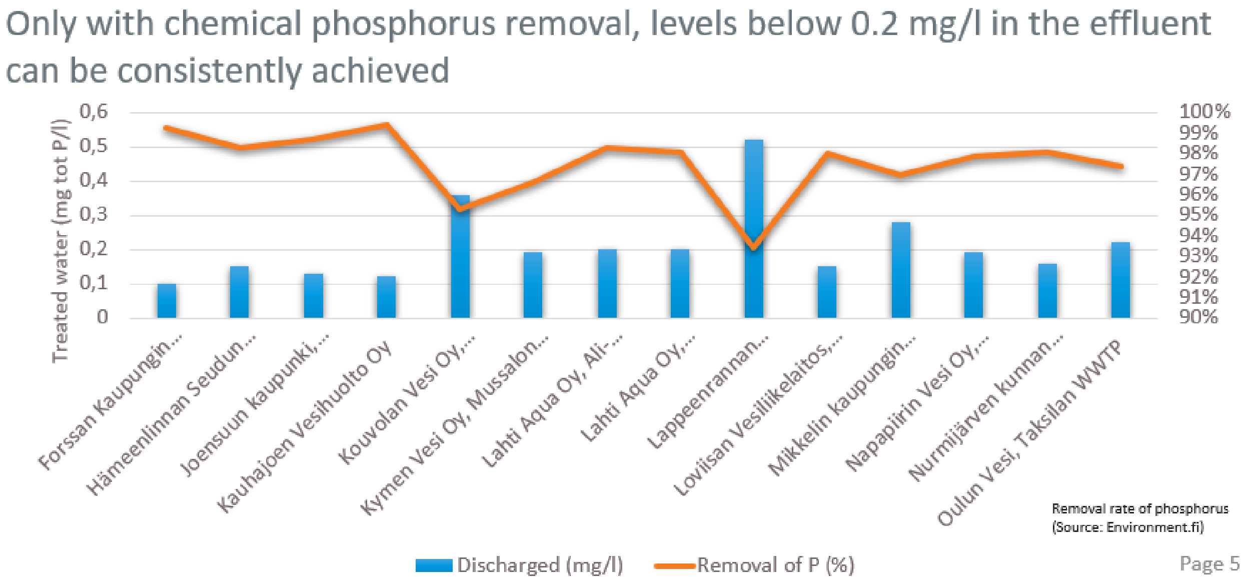
Abstract

INCOPA is the European INorganic COagulants Producers Association. Aluminium and iron salts are essential elements for water treatment, paper manufacturing, fertiliser production and other industries. INCOPA represents 85% of inorganic coagulant production capacity in Europe with 29 producers and 80 production sites.

We have been living in a linear economy since a while. And the phosphorus chain in the wastewater is also following this logic. In this world, we have mined phosphorus, produced phosphorus fertilizers, fertilized the land to produce food and eventually lost the phosphorus with our wastewater into the sludge. Now the world is switching to the circular economy, phosphorus can be recovered from wastewater and recycled as nutrient to the fields, to produce our food, and to close the phosphorus loop.

Inorganic coagulants are part of the circular economy too already for a long time by using large amounts of recycled raw materials in their manufacturing. Now, recovery of phosphorus and inorganic coagulants will fit together. Through recovering phosphorus & inorganic coagulants, we, Inorganic Coagulant European manufacturers, can and will continue to have a great influence on the circular economy.

Phosphorus recovery always start with removal. Chemical treatment, using inorganic coagulants, is the most effective and cost-efficient solution to capture and remove phosphorus from wastewater to make it available for recovery. Coagulants are needed to protect waterways from eutrophication. With chemical phosphorus removal, levels below 0.2 mg/liter in the effluent can be consistently achieved, as demonstrated in the Finnish case below:



Removal rate of phosphorus
(Source: Environment.fi)

Chemical phosphorus precipitation guarantees high phosphorus recovery rates (from 50% to 90%)

By using inorganic coagulants, you can recover:

Phosphorus from sludge:

- Depending on technology, iron or aluminium coagulant can be used and recovered as well
- It can be dosed flexibly to the primary, secondary or tertiary treatment
- Needs chemical extraction of phosphorus or physical separation methods or biological pathways

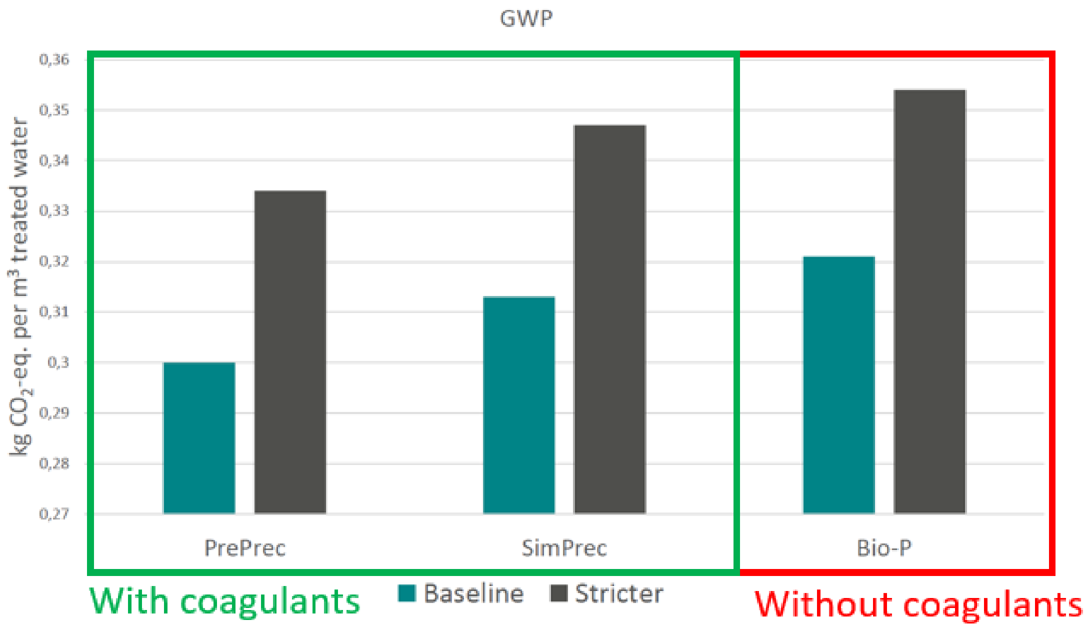
Phosphorus from tertiary treatment:

- The recovery unit is easily installed to the existing wastewater treatment plant
- Coagulant is dosed to the tertiary treatment
- Both iron or aluminium coagulant can be used and recovered as well

Phosphorus from sewage sludge ash

- Normally a very high recovery yield of phosphorus
- Most extraction processes also recover aluminium and iron coagulant
- Mono-incineration of sludge required

In a recent study conducted by IVL, the Swedish Environmental Research Institute, the carbon footprint of 3 different wastewater treatment configurations (pre-precipitation, simultaneous precipitation, Bio-P removal) have been looked at. Chemical pre-treatment (=pre-precipitation) has the lowest Global Warming Potential



As a conclusion, inorganic coagulants used in wastewater treatment:

- Are needed to achieve ultra-low levels of phosphorus discharged in the environment
- Guarantee high phosphorus recovery rate (> 50%)
- Allow higher energy recovery from organic waste and hence lead to a lower carbon footprint
- Have been part of the circular economy for many years

Karel As, Bayreuth University, Germany

I have done my master studies in biotechnology and earth sciences at TU Delft and Utrecht University. Here I worked on the sulfide-based metabolism of cable bacteria. Subsequently I went to Rennes, at the INRA institute where I worked on efficient release of phosphorus from sewage sludge. Currently I am a PhD student at Bayreuth University with Prof. Peiffer working on mechanisms to capture phosphate in lake sediments.

Lake restoration based on Fe Addition

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1) Faculty of Hydrology, University of Bayreuth, karel.as@uni-bayreuth.de
2) Faculty of Earth sciences, Utrecht University

Abstract

In 2000 the European Water framework Directive was installed. This program aims to return all surface waters to good quality. The primary way to achieve this was thought to be a reduction of external nutrient loading. However, many lakes that now have a sufficiently low external load still carry the burden of many decades of external phosphate loading. Phosphate is seasonally released from the sediment and can cause phosphate-caused algae blooms in the overlying water. Such a state can result in a delayed return of water quality parameters. Here we discuss the potential of a waste product from water purification in speeding up this process: iron-oxide coated sands.

Tolulope Ayeyemi, University of Seville, Spain

A Nigerian with a Bachelor of Agriculture degree in Soil Science and Land Resources Management from Obafemi Awolowo University (2013). I worked with the International Fertilizer Development Centre (IFDC), Abuja, Nigeria, as an Agronomist and Master Trainer after my BSc for about 4 years (2013-2017). I obtained my master's degree in Agricultural Science and Resource Management in the Tropics and Subtropics (ARTS) with specialization in soil fertility and phosphorus recycling from the University of Bonn, Germany (2017-2020 Jan). I am now hosted by the University of Seville in Spain for my PhD program under the EU funded P-TRAP project (2020-2023).

Effectiveness of Vivianite as a Sustainable Phosphorus and Iron Fertilizer for Agricultural crops

Tolulope Ayeyemi[1]

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Abstract

Phosphorus (P) as an essential macronutrient is required for the optimum growth of crops. P fertilizers have been produced from the mining of phosphate rock for decades. However, the world reserve of phosphate rock is declining both in quality and quantity; and predicted to last for the next 50 to 300 years. Hence there is a rising need for alternative sources of P for the sustenance of crop production. This research is focused on studying the efficiency of vivianite as a phosphorus (P) and iron (Fe) fertilizer. Vivianite, an iron phosphate mineral ($Fe_3(PO4)_2 \cdot 8H_2O$) contains an average of 32% Fe and 10% P; and is found in predominantly reducing environments as well as in terrestrial systems. It can also be obtained from the precipitation of P from wastewaters with the use of Iron-based phosphate recovery methods from wastewater treatment plants (WWTP) industries. While there have been some studies on the use of vivianite to correct iron chlorosis in certain crops, other few studies have suggested that the concentration of P in vivianite is high enough to make it suitable as a P fertilizer. However, the use of vivianite as a fertilizer product has not been fully explored and there is currently little or no information on its use and behavior as a P fertilizer in different types of soils and crops. In addition to vivianite, this research will also look into the study of other P rich Fe-oxides as a fertilizer and will compare the effect of applying vivianite and other Fe-P oxides with conventional P fertilizers with respect to bioavailability, yield and crop quality. It is hypothesized that the use of vivianite as a fertilizer could help close the phosphorus cycle in agriculture.

Victoria Barcala, Deltares, the Netherlands

Victoria Barcala is a civil engineer, she did her MSc in IHE-Delft where she specialized on phosphorus recovery from agro-industrial wastewater treatment. Currently, she is working on the development of effective mitigation measures to capture phosphorus from diffuse sources. Victoria works at Deltares as an early stage researcher and is a PhD candidate at Utrecht University.

Capturing phosphorus in drained agricultural areas

Victoria Barcala[1,]*, Leonard Osté[1], Thilo Behrends[2], Joachim Rozemeijer[1]
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Abstract

Phosphorus (P) retention mechanisms are needed to improve the surface waters’ quality in drained agricultural areas. Large amounts of P accumulated in the soil during decades of high fertilization, even if fertilizer application is reduced, P will continue to leach for many years. Understanding how the P is transported from the soil to the water bodies is essential to design effective P retention measures. A high-resolution study at the farm scale will be presented and the implication of the results for the design of P retention measures. Finally, future work on large catchment scale P-TRAP systems using Fe coated sand as a P sorbing material will be announced.

Thilo Behrends, Utrecht University, Department of Earth Sciences, the Netherlands

Thilo Behrends research is focussing on biogeochemical processes in natural and engineered systems. His research is mostly process-orientated and involving redox reactions of metals, in particular iron and manganese, and their impact on the partitioning and speciation of oxyanions and trace metals. He is the coordinator of the H2020-MSCA-ITN Project P-TRAP related to using iron-containing by-products from water treatment to reduce fluxes of phosphate to surface waters.

Challenges for trapping and recycling phosphorus from agricultural run-off: Introduction to the P-TRAP project

Thilo Behrends[1], Sylvia Walter, P-TRAP team*

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The context and structure of the ITN project P-TRAP will be introduced. The approaches to reduce the export of phosphate from drained agricultural areas and to convert the phosphate-containing material into useful products for agricultural application will be explained.

Phosphorus (P) as an essential resource for food production is becoming scarce. Its uncontrolled loss from agricultural areas is in conflict with the principles of a circular economy. Enhanced loading of surface waters with P is the main cause for eutrophication and presents a key challenge in meeting the objectives of the EU Water Framework Directive. Understanding and controlling environmental P fluxes therefore is key to target both problems, to develop new methods and approaches to manage environmental P fluxes, and to improve surface water quality.

In the presentation, I will introduce the EU Marie Sklodowska-Curie Innovative Training Network P-TRAP, which has started in March 2019. P-TRAP aims to develop new methods and approaches to trap P in drained agricultural areas and in the sediments of eutrophic lakes. Trapping of P involves the application of iron(Fe)-containing by-products from drinking water treatment. P-TRAP aspires the ideas of a circular economy and aims at recovering the retained P in agricultural systems. Novel microbial technologies will be developed to convert P-loaded Fe-minerals into marketable fertilizers whose suitability will be evaluated. The P-TRAP technologies have in common that they rely on the naturally strong connection between P and Fe and the innovative P-TRAP strategies will be underpinned by process-orientated investigations on the behaviour of P during the transformation of Fe minerals. The latter are key in trapping and recycling of P in agricultural systems and lakes.

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* **P-TRAP team**: As, K., Ammerl T.,Ayeyemi, T., Barcala Paolillo, V., Bolielyi, O., Bovee M.,Branas J., Delgado, A., Eshun, L., ter Heerdt G.,Janneck, E., Krämer, S., Liu, X., Lloyd, J.R., Ma, M., Metz, R., Moegele K., Münch, M., Nenonen, V., Osté, L., Peiffer, S., Reichel, S., Saracanlao, R., Schenkeveld, W.D.C., Smolders, E. , van der Kolk, O., Verbeeck, M., Virosta A., Voegelin, A., Vreman B.-J.,Wesinger R.

Carlo Belloni, Wetsus, the Netherlands

My name is Carlo Belloni, I come from Padova, Italy. I obtained my BC's and MC's degree in Experimental Physics at the Università degli studi di Padova, with a thesis on Nanophotonics. I am now a 3rd year PhD student from TU Delft University, working at Wetsus European center of excellence for sustainable water technology, in Leeuwarden. My research is on Phosphate recovery from surface waters and wastewater through adsorption with iron oxides, using Mössbauer Spectroscopy as the main investigation tool for the adsorbent. I am focusing on the process fundamentals and currently working with iron oxide-based nanoparticles.

Improved recovery of phosphate through manipulation of iron phosphate chemistry using Mössbauer spectroscopy

When working with iron-based compounds, it is of primary importance to understand what we are either harvesting, like precipitate compounds such as vivianite, or working with, like iron-based adsorbents or catalysts. Together with a thorough characterization, an accurate monitoring of the properties of the compound throughout the process is fundamental. Conventional characterization techniques, such as XRD, can fail in identifying iron species, especially when it comes to mixtures or amorphous phases. Other more sophisticated techniques like EXAFS, require an inconvenient sample preparation and use of facilities like synchrotrons, with a scheduled and restricted timeframe. Mössbauer spectroscopy, which is also the highest resolution nuclear technique, is able to overcome these issues, proving to be suited for high accuracy characterization and monitoring. In fact, in previous studies Mössbauer spectroscopy was able to reveal slight phase changes in iron-based adsorbents, after they have been regenerated. Also, commercially available samples were proved to be different from what the provider claimed. The current work, dealing with doped iron oxide adsorbents for phosphate, showed the homogeneity of the doping and the consequent new features of the material. The aim of this presentation is to show how Mössbauer spectroscopy constitutes a valuable option and fundamental tool for iron-based compounds investigation.

Sarah Bluteau, McGill University, Canada

Sarah graduated with a B.A.Sc in chemical engineering at the University of Ottawa, Canada. She completed her Masters of Engineering in Mining and Materials Engineering at McGill University, Canada, where she is now a Research Assistant. Sarah's research focusses on dissolving phosphate from anaerobic biosolids produced by a municipal WWTP, and phosphate precipitation as a useful P-fertilizer. She is currently optimizing and scaling up her P-recovery process to a larger lab scale.

Phosphorus recovery from iron phosphates in biosolids with sodium sulphide

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This work investigated the dissolution of inorganic orthophosphate (o-Pi) within a mixture of biosolids resuspended in centrate by addition of sodium sulfide nonahydrate. The ratio S:"total phosphorus" (TP) molar ratios investigated were 0 to 5; a 4:1 S:TP ratio increased the o-Pi concentration of a 10 % solids-centrate mixture from 0.1 to 43 ± 1 mM o-Pi after 11 h at room temperature. This dissolution represents 42 % of the total P in the biosolids.

Abstract

The potential for dissolving the o-Pi component of anaerobic biosolids produced by the Ottawa (Canada) municipal wastewater treatment plant with sodium sulphide ($Na_2S \cdot 9H_2O$) was tested. The measured total phosphorus (TP) concentration in the biosolids after an oxidative and acidic digestion was 0.4 mM P/dry gram biosolids [1]. The concept was to reduce the soluble iron activity (α) by its association with dissolved sulphide (S^{2-}) at low Eh (~-650 mV) and alkaline pH (~11) [2]. This would reduce the ion activity product (IAP) of vivianite ($Fe_3(PO_4)_2\ 8H_2O$) [3] (Eq. 1). This reduced $IAP_{vivianite}$ would reduce the vivianite saturation state ($S_{vivianite}$), with the $Ksp_{vivianiate}$ (Eq. 2).

Eq.1

$$IAP_{vivianite} = ¿$$

Eq.2

The solubility product of FeS was theorized to be the thermodynamically favoured iron salt in this low Eh, high pH system, therefore driving vivianite dissolution.

Following previous work [3-5], a range of S^{2-}:TP ratios were studied for different (2-15 %) percent solids values in batch tests of anaerobic biosolids mixed with centrate for 72 h. A trend of increasing dissolved o-Pi with increasing S^{2-} concentration was observed (Fig. 1).

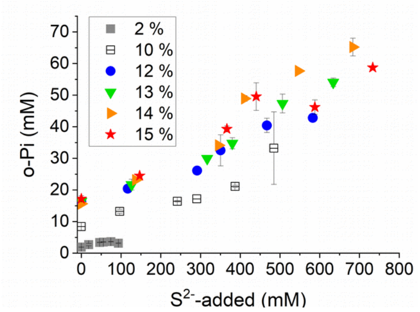
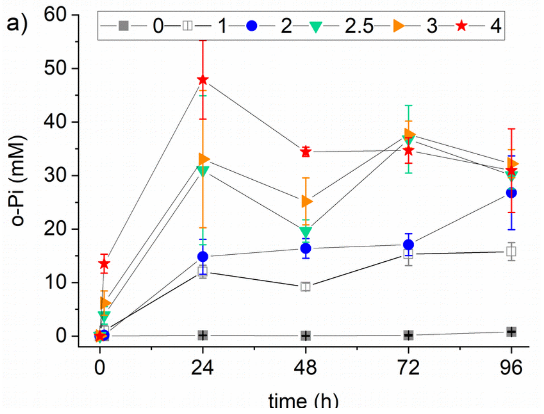


Figure 1: [o-Pi] as a function of [S^{2-}] and % solids, 72 h.

An optimization of viscosity (10 % percent solids) was carried forward to study the effect of contact time and S^{2-}:TP ratio. Figure 2a presents the increase in o-Pi. The Eh value of the control (no S^{2-}-treatment) sample after 4 days was -230 ± 10 mV, while the Eh values of the S^{2-}- treated samples were of increasingly negative values (-430 - -600 ± 10 mV with increasing S^{2-}:TP ratio. After 4 days the pH value of the control sample was 7.6 ± 0.2, and increased from 9.2 – 11.5 ± 1. The maximum dissolved o-Pi was 48 ± 20 mM, representing 42 % of the theoretical TP concentration (114 mM).

Figure 2b presented the dissolved Fe concentration (ferrozine assay) for the sample groups after 4 days of shaking in sealed tubes at room temperature.



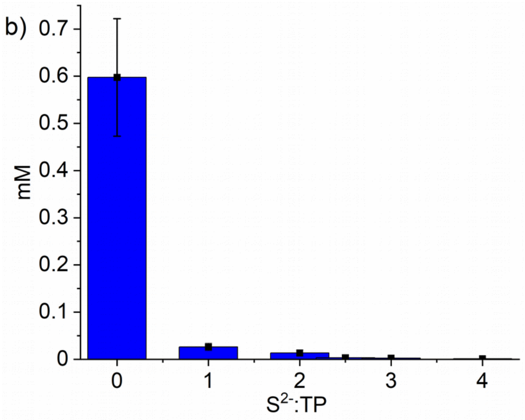
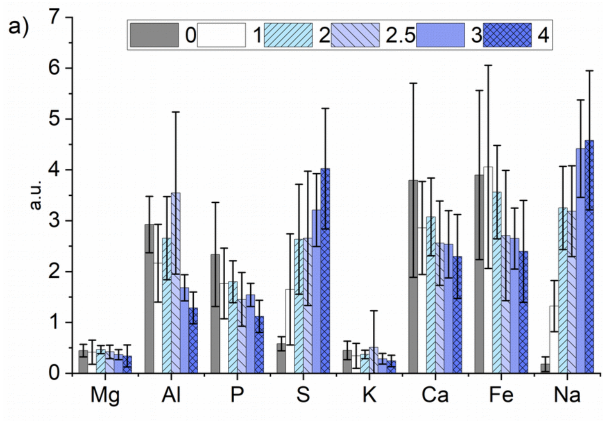


Figure 2a: o-Pi concentration as a function of time and S^{2-}:TP rato.
Figure 2b: Dissolved Fe concentration as a function of S^{2-}:TP ratio after 4 days.

The solid treated products were darker than the biosolids, suggesting iron sulphide precipitation. The solid products of these experiments were dried and analysed with EDS (Fig. 3a) and powder X-ray diffraction (p-XRD, cobalt source (Fig. 3b).



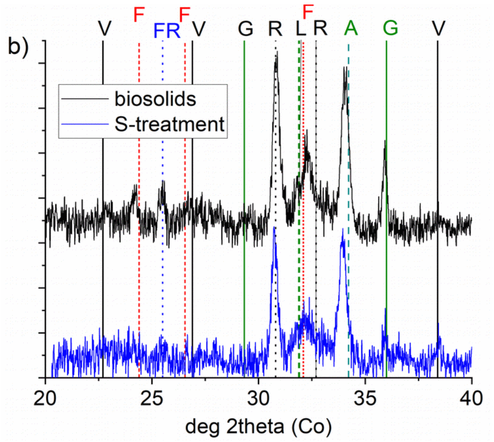


Figure 3a: EDX data of solids as a function of S^{2-}:TP ratio, after 4 days.
Figure 3b: XRD (Co-source) of biosolids (upper, black) and 4:1 S^{2-}:TP (lower, blue).

Although EDX and p-XRD data are not quantitative, an increase in S and Na in the solids was detected. The Al and P content decreases are plausible, and supported by the reduction of major peak intensity (in °2θ Co source) for ferrorosemaryite (FR): $NaFe^{2+}Fe^{3+}Al(PO_4)_3$ (notable peak 25.5 °2θ) $FeFe_2(PO_3OH)_4(H_2O)_4$ (F): (major peak 32.1, also 24.4 and 26.5 °2θ), rockbridgeite (R): $Fe^{2+}Fe^{3+}{}_4(PO_4)_3(OH)_5$ (major peak 32.7 °2θ, notable peak 30.8 °2θ), arrojadite (A): $KNa_4Ca(Fe,Mn)_{14}Al(OH)_2(PO_4)_{12}$ (major peak 34.2 °2θ Co), graftonite (G) $(Fe,Mn,Ca)_3(PO_4)_2$ (major peaks 36.0 °2θ) and laueite (L): $MnFe_2(OH)_2(PO_4)_2(H_2O)_6.2H_2O$ (notable peak 31.9 °2θ). The vivianite major peak (15.3 °2θ) was obscured by fluorescence. The iron sulphide solids are likely amorphous. It is concluded that a 4:1 S^{2-}:TP for 10 % solids of an anaerobic biosolids/centrate mixture dissolved approximately 42 % of the TP after at most 24 h.

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Hans Christian Bruun Hansen, University of Copenhagen, Denmark

Hans Chr. B. Hansen is professor in Environmental Chemistry at University of Copenhagen. He is working with phosphorus adsorption, mobilization, and retention in soils, and filter solutions to trap phosphorus in agricultural drainage water. In most of his research phosphorus and iron biogeochemistry has been combined. Main results on phosphorus mobilization in lowland soils, and new iron oxide based filter materials.

Iron oxide coated porous filter material for agricultural drainage water

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2) DiaPue AB, Mellangårdsvägen 4, 230 53 Alnarp, Sweden

New filter material has been develped that combines high hydraulic conductivity with efficient sorption of phosphorus

Abstract

Agricultural drainage water is a major source of phosphorus to surface and coastal waters. Phosphorus concentrations in agricultural drainage water typically range between 0.1 and 1 mg P/L - and thus above eutrophication thressholds of 0.05 mg P/L. Higher drainage water concentrations may be seen for anoxic lowland soils where phosphorus is mobilized from dissolving iron(III)oxides. Phosphorus in drainage water may be retained by filters, which are placed in the drainage system, typically at end of drainage pipe before the drainage water is discharged to recipients. Drainage water filters are compact and cost efficient solutions, and the phosphorus retained can be reused. Because of huge volumes of drainage water, water flows are high and residence times short. Hence, filter material should consist of larger particles (> 2 mm) with high phosphorus bonding affinity and capacity, and the material should be physically stable to ensure long term use.
A new phosphorus filter material has been developed to fulfill these demands. The material comprise 2 - 4 mm calcined diatomaceous earth (CDE) particles which have been coated with iron oxide (Fig.1). The granules have an internal porosity higher than 50 % with a bimodal pore size distribution (pores < 1 um and pores > 100 um). The granules are coated with iron oxide by first soaking the material in iron(III) chloride solutions followed by precipitation of iron(III) (hydr)oxides with alkali. This produces a material amended with 3 - 10 % of Fe, that both have high hydraulic conductivity and efficient phosphorus retention. When packed in filter columns phosphorus in drainage water initial sorbs to outer sorption sites on the CDE granules and next migrates into the interior of the granules.

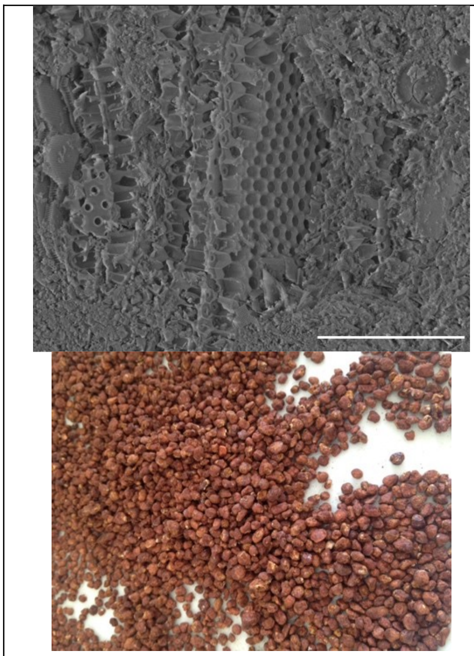

Figure 1: Calcined diatomaceous earth showing a SEM image of the raw material (scale bar 50 um) (top) and the iron oxide coated filter material (bottom).

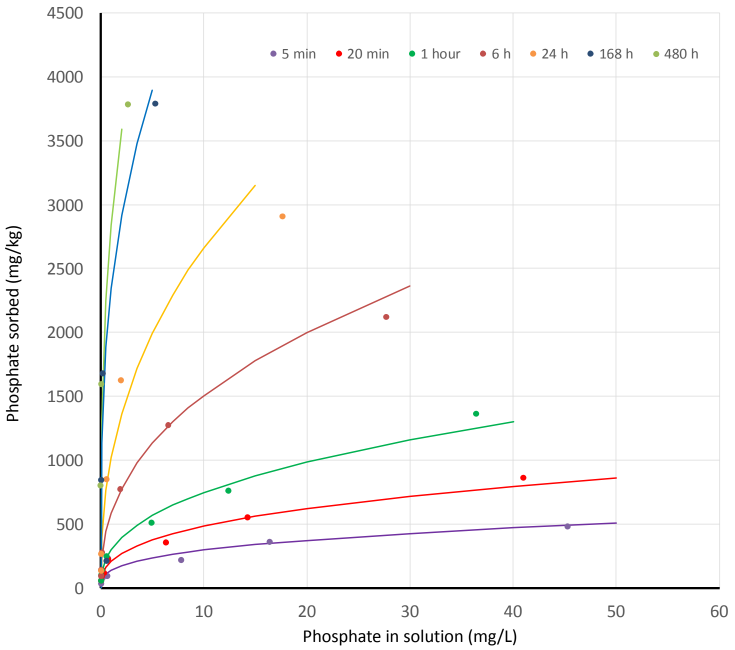

Figure 2: Sorption isotherms for iron oxide coated diatomaceous earth (approx. 4 % of added iron; BET 68. m^2/g). Sorption data and corresponding Freundlich fits refer to exposure times between 5 min and 480 h.

The filter material has high hydraulic conductivity (K_{sat} 0.0092 m/s). Phosphate sorption capacity and affinity inceases strongly with time (Fig. 2) and corresponds to the sorption properties of hydrous ferric oxide (ferrihydrite). Column studies show that during prolonged use filters can sorb > 5000 mg P/kg at solution concentrations of 1 mg P/L. The robust filter material allows for regeneration enabling phosphorus recycling.

References:

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Lyngsie, G.; Kjærgaard, C.; Hansen, H.C.B. (2016) Metal oxide coated diatomite aggregated and use thereof as adsorbent and fertilizer. Patent WO 2016/041563 Al. https://patentimages.storage.googleapis.com/62/6d/30/c24ef710d276bf/WO2016041563A1.pdf

Cinta Cazador, FERTIBERIA S.A., Spain

Her background is Chemical and Environmental Engineer. She has been working in Fertiberia since 2005 in Research and Development Area. She is in charge of the design, implementation, commissioning and plant adaptations for new processes and products, from laboratory, pilot plant, demo plant and industrial plant. Nutrient recovery from biobased wastes as well as valorisation of residues in the fertiliser industry are also part of her main roles.

The specific case of recovered vivianite as fertilizer

Cinta Cazador[1], Javier Branas[2]

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Vivianite has demonstrated its efficiency to prevent iron chlorosis in calcareous soils in crops such as olive trees, vineyard, kiwifruit, pear trees, peach trees, etc.
Vivianite can be obtained by precipitation of phosphorus in waste water (EP 266 6759 B1).
Microcosm and field trials have been carried out by Fertiberia since 2011, in which vivianite and vivianite mixed with NPK have proven their efficiency in plant nutrition and iron chlorosis prevention.

ABSTRACT

Iron chlorosis is a chlorophyll deficiency in leaves caused by an iron (Fe) nutrition disorder. Iron chlorosis affects crops of great socioeconomic significance, such as sunflowers, chickpea, vines, peach, kiwifruit, etc., but above all it affects the olive trees (Del Campillo and Torrent, 2005). Vivianite applied to calcareous soils is capable of preventing iron chlorosis in lupins and chickpeas cultivated in pots under controlled conditions (Eynard et al.,1992), in pear trees cultivated in the field by making use of traditional agricultural practices (Iglesias et al., 1998) and in olive groves over at least 3 years (Rosado et al., 2002).

Vivianite has been obtained following the methodology explained in the European Patent EP 266 6759 B1 (owned by Incro S.A. from Fertiberia Group).

Pot trials in controlled conditions have been carried out. One of them was made in calcareous sand with cucumber as a crop under study. Different amounts of vivianite were applied in the soil in comparison with the recommended iron chelate dosage.

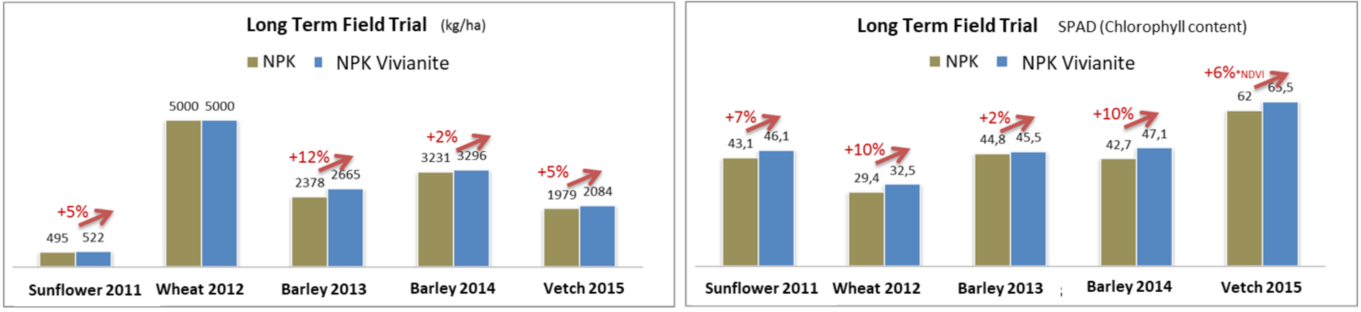
The results showed that there were not significant differences in terms of growth and SPAD index between the plants treated with vivianite and the ones treated with iron chelate. The efficiency against iron chlorosis of the vivianite obtained with the proposed methodology has been proven. Table 1 shows the main results released after this trial:

Table 1. SPAD index, dry matter and fresh matter obtained after the trial:

Treatment		SPAD	DM total (g)	FM AP (g)
T1	no Fe	8 b	1.3 c	5.5 c
T2	Fe-EDDHA	47 a	3.0 ab	11.5 ab
T3	0,25 g viv/kg soil	45 a	2.5 b	11.2 ab
T4	0,50 g viv/kg soil	44 a	2.7 ab	11.6 ab
T5	1,00 g viv/kg soil	44 a	3.6 a	12.9 a
T6	1,50 g viv/kg soil	48 a	2.7 ab	12.3 a

Furthermore, a long term field trial was performed for 5 years in a high calcareous area in Valladolid (Spain) in different crops every year (rotation), in order to demonstrate that the application of vivianite incorporated in the NPK fertiliser improved both the plant nutrition and chlorophyll content (directly related to iron chlorosis). The plot was the same in every year, and the same treatment (except for vetch in 2015, in which there was not vivianite application) was applied in each experimental unit once a year. Figure 2 shows the final results after the 5 year trial:

Figure 1. Crop yield and SPAD index for NPK (8-15-15) and NPK with vivianite (8-15-15 with 0,5-1% Fe)



The results showed that there was a general increase in crop yields and SPAD index, which validated the use of vivianite incorporated within the NPK fertiliser matrix as a methodology of improvement of iron nutrition in plants. In 2015 despite vivianite was not applied the effects of it still remained as an amorphous iron salt in the soil, which is susceptible for being absorbed by the roots of the plants when it is demanded by them.

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Marie-Line Daumer is a Research Engineer at INRAE (ex IRSTEA). She has been working on P phosphorus (P) recovery from sludge and animal manure for many years. She was involved in the NEWFERT project (BBI-JU) to produce secondary P raw material from pig manure for fertiliser industry and is now involved in the Phos4You project (Interreg NWE) to recycle P but also Fe from sewage sludge after dissolution by a biological process.

Biologic routes for release of phosphorus from iron or aluminium compounds in sewage sludges

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Acknowledgment: French Agency for Biodiversity (PhoSTEP-project); Interreg NWE, Phos4You project

Depending on the technology used to remove phosphorus (P) from wastewater, it could be either as bio-P or as Iron or aluminium salts in sewage sludge. Bio-acidification is a promising process, more efficient with less environmental impacts than chemicals, to increase P recovery from sewage sludge by dissolving up to 75% of P even with sludge coming from Iron or aluminium P removal processes (Guilayn et al., 2016). Our objective is to determine how the forms of P and Iron in sludge, and so, how the P removal process upstream could impact the bio-acidification efficiency.

Material and methods

Two experimental sets were performed. In the first one 20 sludge, from 10 different WWTP, using 5 different P removal processes, sizing from 10 000 to 620 000 p.e were tested for their biological phosphorus dissolution potential (BPDP) (Braak et al., 2015). In the second one, ten sludge, from 5 different WWTP, using 3 different P removal process were tested for the BPDP. The forms of P and Iron were analysed before and after the bio-acidification. To be able to analyse both P and Iron forms in sludge a method was developed combining three sequential extraction methods. The total P, Ca, Fe, Mg and Al content of each extract was measured by MP-AES after acid digestion. The mass balance was calculated as the ratio between the sum of the fractions and the total amount in sludge.

Results

The first experimental set shown that the metal content in sludge was not always the one that could be expected from salts used for P removal upstream. That could be due either to the city network management for odor control or to sludge coming from other WWTP. The BPDP was between 50 and 70% when the sludge was coming from biological P removal (EBPR) or EBPR + $FeCl_3$. Between 30-50% were observed in sludge coming from the same WWTP using EBPR + Al salts. Less than 10% was observed in sludge using only Al salts for P removal (Figure 1). No clear relation with pH reached at the end of the bio acidification tests or the metal/P ratio was demonstrated. The second experimental set has shown that the P linked to Fe^{3+} was dominant (30-

60%) in all the sludge. P linked to Fe^{2+} was more than 20% only in 2 sludge coming from WWTP combining EBPR and $FeCl_3$ for P removal (Figure 2). Fe^{2+} was dominant in sludge coming from WWTP combining EBPR and FeCl3 and in primary sludge when chemical P removal was performed in the primary sludge.

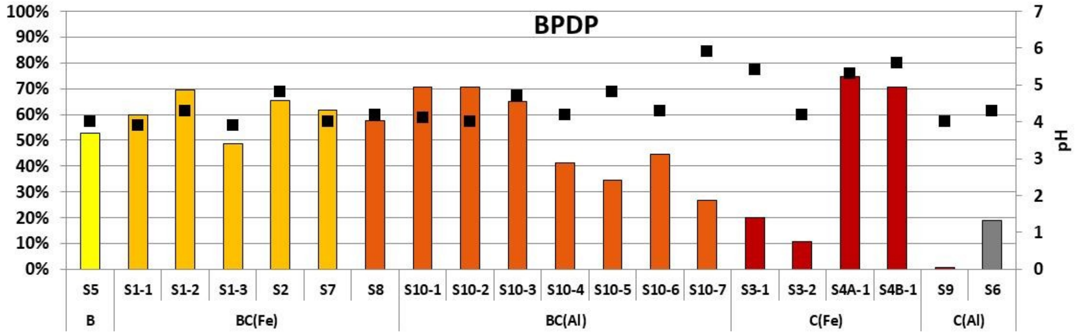

Figure 1 : Biological phosphorus dissolution potential of sludge coming from different P removal process. B: EBPR; C: chemical; BC:EBPR+chemical; Fe: Iron salts; Al: Aluminium salts

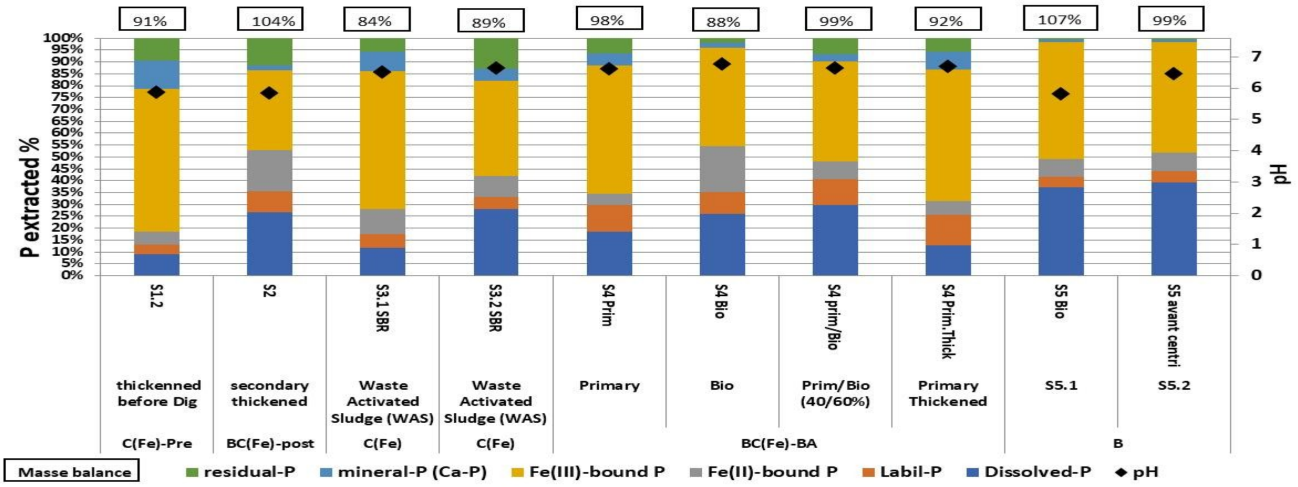

Figure 2 : Forms of phosphorus in sludge coming from different P removal process. B: EBPR; C: chemical; BC:EBPR+chemical; Fe: Iron salts; Al: Aluminium salts

The results on the forms of P and Fe impacted by bio-acidification are under validation and will be available for the workshop.

Conclusion

Forms of P and Fe in sewage sludge are strongly depending on the P removal process in WWTP but also on how this process is managed. A little change in P removal processing could significantly increase the P dissolution and so the P recovery rate by bio-acidification.

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Antonio Delgado, University of Sevilla, Spain

Professor of Agronomy; research activity focused on soil fertility and management, fertilization management (mainly P, Fe, Zn), and the interaction of organic matter and microorganisms with plant nutrients. Collaboration in studies on the environmental impact of fertilization, soil amendments and soil salinization. He participates in the project h2020-P-TRAP.

Iron phosphate as fertiliser on Mediterranean soils?

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Abstract summary

Iron (II) phosphates may be obtained from water purification processes. The compound contains Fe and P, two essential nutrients form plants and has been proved to be an effective Fe fertilizer for control of Fe deficiency chlorosis in calcareous soils typical in Mediterranean environments. However, there are no evidences on its efficiency as P fertilizer in this type of soils. The Fe fertilizer effect depends on the dissolution of vivianite particles in rhizospheric environment and on the precipitation of poorly crystalline Fe oxides which are sources of Fe for plants. The disruption, dissolution and oxidation of vivianite is supposed to be related to the potential mobilization and uptake of P by plants. Thus, it can be supposed that vivianite may act as a P fertilizer. In addition, the potential of vivianite as P fertilizer will depend not only on economic considerations but also on its long-term ability to release bioavailable Fe and P, which would contribute to make fertilization more environmentally friendly. The assessment of the P fertilizer effect of vivianite is one of the objectives of the H2020-P-TRAP project.
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Alfred Edlinger, MITechnology, Austria

Alfred Edlinger is leading an Austrian SMU "MITechnology". Company is dedicated to develope new thermochemical processes for the CO_2-minimized circular economy. Target is to produce zero wastes and marketable products like cements, metallalloys, metallconcentrates, phosphorus, zinc, syngas (exergy optimisation).More than 600 IP´s demonstrates the developments (see references). Several awards like the Austrian scientific price, Austrian state awards, etc.for MITechnogy's developments.

FerroPhos

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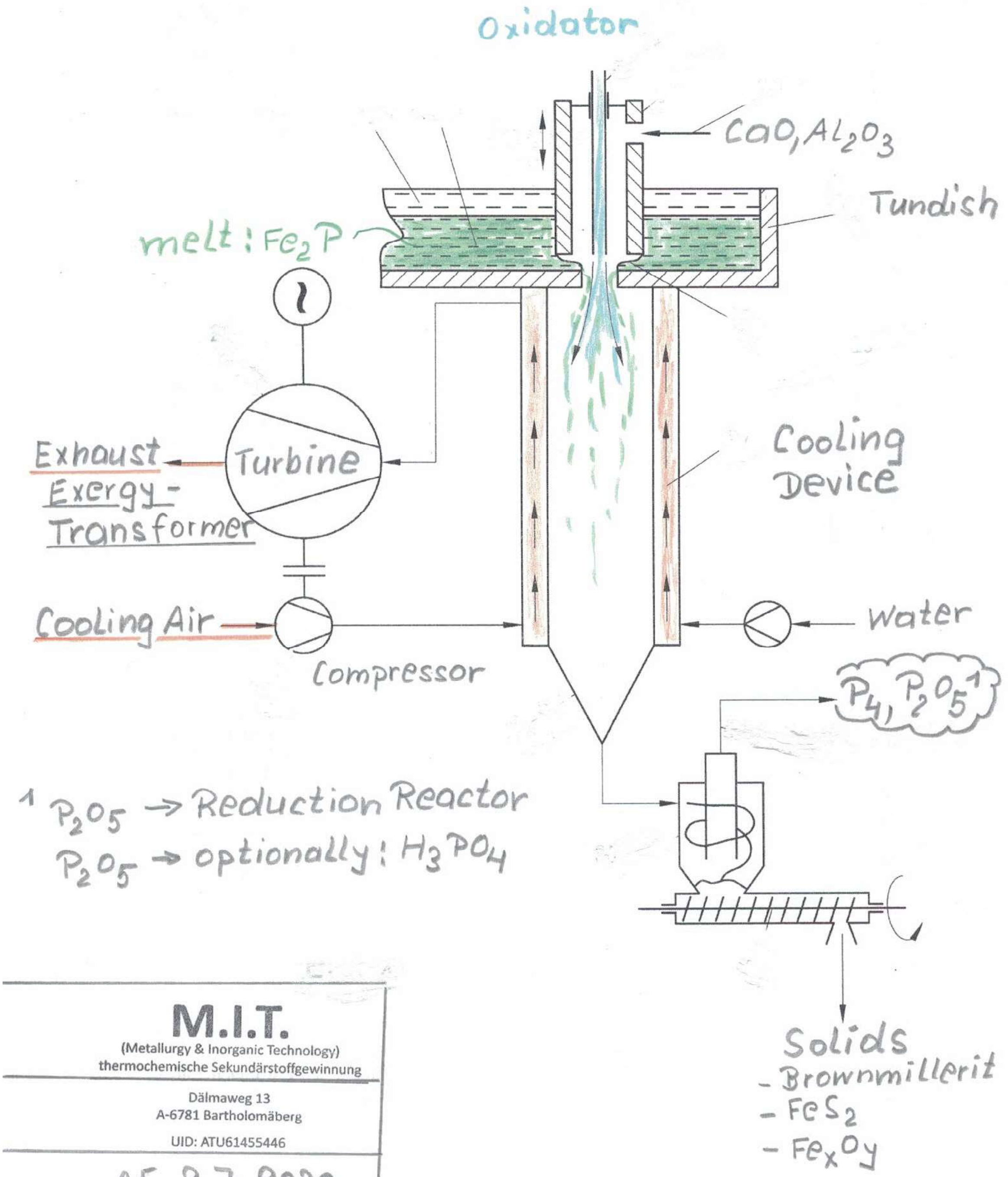
Thermochemical conversion of ferrophos alloy(s) in elementary phosphorus and ironcompounds

FerroPhos is an "End of Pipe Process" and separates elementary phosphorus (P4) completely from iron. Remaining ironcompound is simultaneously converted in a useful ironcompound like Brownmillerit (2CaO.(Al,Fe)2O3) for Portlandclinker burning.
Ferrophos alloy is produced as a byproduct by carbothermical reduction of phosphate containing materials like Apatite (Woehler Process), Sewage sludge/ash, bone meal, metallurgical slag, waste electronics, waste Li-battery and others.
Products of the FerroPhos Process are P4 or P2O5, cement additives , synthetic iron ore. Ferrophos alloy (ironphosphides) are a significant loss on P4. However there is also a small demand for special steel and cast iron (Iron Foundry).
FerroPhos is an oxidation process with oxygen, sulphur and sulphur containing agents in the melt phase and worldwide protected.

- Brownmillerit formation : ironphosphides to be added with CaO and Al2O3 containing carriers as well as oxygen containing gases leads to gaseous P2O5 and Brownmillerit melt, a valuable mineral to be added as sintering aid for Portland Clinker formation. Brownmillerit has no affinity to P2O5.
- Ironoxide formation :simple oxidation with O2 – containing gases of ironphosphides leads to P2O5 and ironoxide. Total separation on special thermochemical conditions is possible. Ironoxide can be used as synthetic iron ore (e.g. can be used for Blastfurnace or Direct Reduction processes or also for Clinker production).
- Sulfidisation : Treatment of ironphosphide melt with S or sulfur containing agents leads to elementary phosphorus and ironsulfide. Ironsulfides shows no affinity to P2/P4. Ironsulfide can be calcinated under oxidation conditions to ironoxide and SO2 . SO2 can be redirected to the FerroPhos – Process and be used for the Oxidation of ironphosphides.
- Sulfitisation : Ironphosphide oxidation with SO2 leads to P2O5 and ironsulfide.

The partly resulted gaseous P2O5 (see above) can be easily reduced to elementary Phosphorus via a newly developed reduction reactor concept (RR). RR is using biomass and H2 for reducing P2O5 to elementary phosphorus, beside CO and H2/H2O, which can be used also via water gas reaction, e.g. for a prereduction step. No phosphine formation .
Exergy for pyrolysis of biomass can be generated by its recuperation by a special melt granulation step, called "Chem2Gran" , under development by MITechnology.
So in total the FerroPhos process concept is CO2 – neutral.
For the exothermic oxidation of ironphosphide melt an Oxidation Reactor concept was developed and tested – see attached sketch and Video ("FerroPhos – FlashOxidator", DGM -project)

FerroPhos - FlashOxidator
(Sketch)



1 P_2O_5 → Reduction Reactor
P_2O_5 → optionally: H_3PO_4

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AE, 3.7.2020

References :see Espacenet, Database of the European Patent Office https://worldwide.espacenet.com/searchResults?submitted=true&locale=en_EP&DB=EPODOC&ST=advanced&TI=&AB=&PN=&AP=&PR=&PD=&PA=&IN=alfred+edlinger&CPC=&IC=

Jürgen Eschment, PARFORCE Engineering & Consulting GmbH, Germany

Member of Executive Board of PARFORCE, responsible for Finance, Strategy, HR and Legal Affairs. PARFORCE is a Spin-Off from the Freiberg University of Mining and Technology, Saxony, that offers the PARFORCE-Technology amongst others to municipal waste water treatment plant operators, sewage sludge incinerators as well as to the phosphate industry to recover phosphoric acid from residues containing P. Before joining PARFORCE Mr. Eschment held several senior management positions in the energy and water industry as well as being an independent strategy consultant.

Phosphoric acid recovery from phosphorous-containing materials

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PARFORCE is a patented wet-chemical, power-to-chemical process to gain phosphoric acid from a wide range of different primary and secondary materials containing phosphorous, making it to a viable solution to meet legal P-recovery obligations. Major difference of PARFORCE to current best available technologies for gaining phosphoric acid is by implementing an electro-dialysis to separate calcium instead of using sulphuric acid to precipitate calcium as gypsum. Advantage of the electro-dialysis is, that it also separates other mono- and divalent ions, such as Magnesium and most toxic heavy metals, from acid leached materials containing phosphorous.

Phosphorous is a critical resource for European countries, as Europe has almost no natural deposits of its own. Some countries such as Switzerland and Germany have already passed legislation to recover Phosphates in future from certain waste streams, such as sewage sludge or sewage sludge ashes. Austria is in preparation of such legislation.

Major challenge in recovering P from sewage sludge is, that most waste water treatment operators rely on iron or aluminium salts in their waste water treatment to eliminate P. Furthermore, iron and aluminium based additives are used to improve anaerobic sludge digestion or to prevent filamentous bacteria from interfering with water treatment processes. Molecular mass ratio of iron and aluminium to phosphorous is often above 1,2 so that phosphorous is almost completely bound as iron and aluminium phosphate making in general recovery from sewage sludge almost impossible and from ashes complex.

The PARFORCE-Technology gains phosphoric acid from materials such as rock phosphate, di/tri-calcium phosphates from industrial residues, bone meal ashes as well as from Struvite and sewage sludge ashes. The technology consists of four major process steps, which are the same for any material processed (PARFORCE-Core). The four steps are:

1) Leaching with diluted acid to mobilise P. Either hydrochloric acid or nitric acid can be used for leaching. By using different acids either nitrate or chloride salts result as a by-product, which may in one way or the other be more favourable.

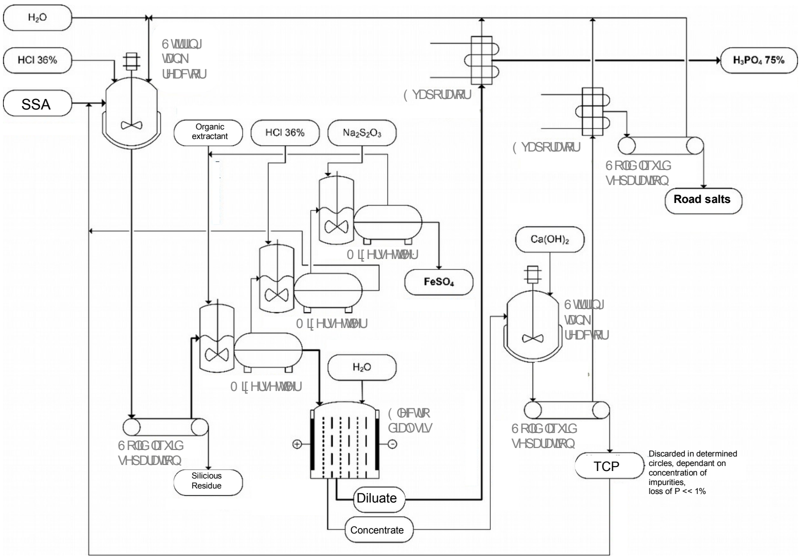
Solid liquid separation to eliminate siliceous residues from the leaching solution.

Electro-dialysis to separate mono-/divalent ions (Cl^-, Na^+, K^+, Ca^{2+}, Mg^{2+}, Cu^{2+}, Pb^{2+}, Cd^{2+}) from the phosphoric acid. The ions are transported into a salt solution (concentrate flow in Picture 1), which can be processed further, i.e. to road salts or to recover leaching acid

Concentration of H_3PO_4 (diluate flow) to tradeable standard of 75 … 85% by vacuum evaporation.

Additional steps are added for certain input material. When Struvite is processed, a calcination step is set before acid leaching to eliminate any adhesive organic matter and to eliminate and recover ammonia. The resulting salt solution from the electro- dialysis will be $MgCl_2$, which can directly be used as a magnesium source for Struvite crystallisation.

When P is recovered from sewage sludge ashes, a solvent Fe/Al-extraction step using mixer-settler units is integrated prior to the electro-dialysis to prevent scaling of the membranes (Picture 1). Re-extracting Fe/Al from the organic extraction medium with hydrochloric acid delivers Fe/Al containing precipitation salts.



Picture 1: Simplified process outline for processing SSA to produce phosphoric acid and road salts as a by-product.

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Lordina Ekua Eshun, University of Manchester, United Kingdom

Lordina Eshun is a motivated Ghanaian female with an MSc degree in Environmental Sciences (specialized in Env'tal Technology) from Wageningen University in the Netherland. She worked with Wetsus (Leeuwarden, Netherland) for both her master internship and thesis (Removal of phosphonate-based antiscalant by electro flocculation and Full-scale increased iron dosage to stimulate the formation of vivianite and its recovery from digested sewage sludge respectively). She is currently focused on developing a biotechnological approach to recover phosphorus via vivianite formation (or other useful Fe-P materials) which can be tested for their effectiveness as Fe and P fertilizers in agriculture.

Microbial Technologies for converting phosphate-rich Fe(III) oxides

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Abstract

Iron-reducing bacteria including *Geobacter sulfurreducens* and *Shewanella putrefaciens* have been used to catalyze the biotransformation of Fe(III) oxides and oxyhydroxides to Fe(II)-containing functional nanomaterials such as magnetite and siderite. Vivianite, a phosphate-rich Fe(II) mineral ($Fe_3(PO_4)_2 \cdot 8H_2O$), has also been reported to form as a secondary mineralization product under reducing conditions when phosphate is present. Phosphorus (P), as a plant-limiting macronutrient, is also the major contributor to eutrophication when it reaches surface water bodies. Thus, the development of an innovative strategy for trapping, recovery, and management of P is crucial. This project, therefore, seeks to trap phosphate in a bioavailable product, by optimizing the bioconversion of phosphate-rich Fe(III) oxides to vivianite (or related biominerals), which can then be tested for effectiveness as a fertilizer in Fe and P deficient soils (via secondments to industrial and academic partners in Spain: FERTIBERIA and the University of Seville). The sorption of phosphate onto Fe(III) oxyhydroxides and the subsequent bioconversion to vivianite and other useful products will be investigated initially by using synthetic substrates, and then waste materials (e.g. Fe(III)-containing waste sludge with microbial inocula in bioreactors). The project will include cutting edge microbial/DNA analyses in Manchester, and advanced spectroscopic characterization (with support from EAWAG, Switzerland).

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Business Development Manager – New Technologies
Proprietary Technologies Development Division

Dealing with iron with Pho4life Technology

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Abstract summary

Sewage Sludge Ash (SSA) is the residue obtained during the combustion of dewatered sewage sludge in an incinerator. The major chemical constituents of SSA are oxides of silicon, aluminium, iron, calcium, magnesium and phosphorous. In this complex matrix, the quantitative separation of iron and phosphorous is one of the most important challenges.

ZAR Foundation and Técnicas Reunidas have jointly developed Phos4life process, financed by the Swiss Canton of Zurich and Técnicas Reunidas. Phos4life technology offers a phosphorus recovery path from sewage sludge ash in the form of technical grade phosphoric acid, a high-performance chemical reagent. Additionally, this technology, if required, can deal with iron transforming it into ferric chloride 40% solution.

Phos4life technology offers two different ways to deal with iron and phosphorous in the leaching stage:

1. The dissolution of both elements, putting the SSA in strong acid conditions which achieves leaching efficiencies close to 98% for P and 65% for Fe.
2. The selective leaching of P under soft conditions, which puts around 90% of P in solution and leaves more than 93 % of the total Fe in the solid.

The final selection of the best approach depends on the specific raw material, environmental requirements of the countries and market opportunities.

The process (figure 1), first developed at lab scale, has been tested and further optimized in a pilot plant installed at TR's Technology Centre. Phos4life is made up of 8 operational units (three of them devoted to ferric chloride production), so that reagents consumption and residues production are minimized while phosphoric acid product recoveries are maximized reaching >90% of global phosphorous recovery. Alternatively, if an additional leaching stage is added, also a ferric chloride 40% solution can be produced obtaining a global Fe recovery efficiency >50%. Moreover, the efficient depletion of heavy metals from the mineral matrix enables recycling this material in construction material plants as it is formed by gypsum for the most part.

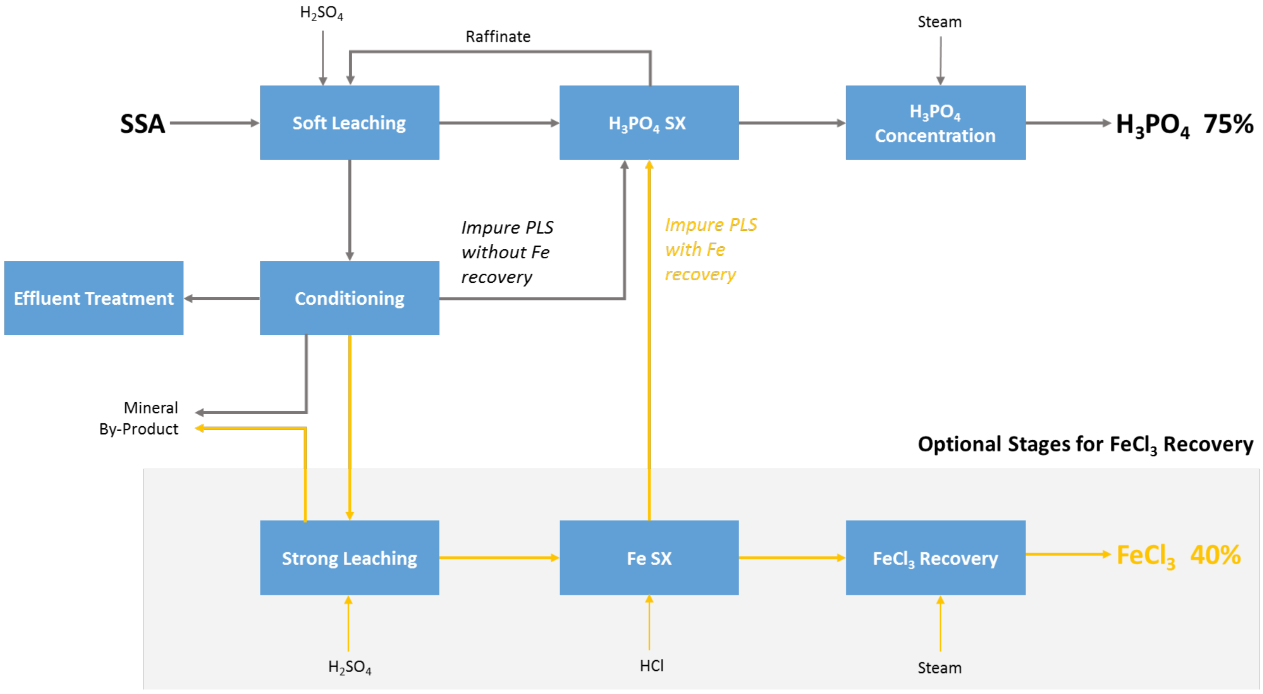

Figure 1: Block diagram of Phos4life process

Phosphorus solubilization mainly takes place at the soft leaching stage unit. In this unit sulfuric acid is added as leaching agent. In addition of the phosphorus reaction, other elements present in the raw material are also partially dissolved.

After a Solid/Liquid separation, the liquid obtained in this soft leaching passes through a conditioning stage which helps to transform the impure leaching solution into an impure phosphoric acid pregnant liquor (PLS) able to be send to phosphoric acid solvent extraction. This conditioning stage differs if ferric chloride recovery is present.

Depending on the process requirements, the solid produced in the soft leaching can be sent to battery limits for its recycling as construction material or to a strong acid leaching stage in which extra sulfuric acid is added to deplete phosphorus from the solid and achieve iron solubilization.

The resulting liquid stream enriched in both elements enters iron solvent extraction unit to selectively separate iron from phosphoric acid in two different aqueous streams: an iron (III) chloride loaded stream and an impure solution of phosphoric acid (PLS).

The iron chloride stream, also enriched in hydrochloric acid, enters the HCl purification unit producing clean hydrochloric acid, ready to be reused in the circuit and a pure iron chloride solution, ready to be concentrated in the corresponding $FeCl_3$ concentration unit. The outcome of this unit is an iron chloride solution that can be directly commercialized to water treatment plants.

Regarding the impure phosphoric acid solution (PLS), it is delivered to a multi-stage purification route, using a H_3PO_4 solvent extraction unit to firstly separate detrimental impurities from the acid and secondly to concentrate phosphoric acid yielding a pure phosphoric acid solution. The clean solution is finally sent to a final purification unit where sulphate and arsenic direct removal is performed yielding a pure final acid product. Further concentration of this stream produces a final technical grade phosphoric acid 75% devoid of the impurities present in the sewage sludge ash inlet.

During the phosphoric acid solvent extraction, also a dirty solution is produced. Part of this solution is send back to the soft leaching, to reutilize the sulfuric acid containing, and minimize the phosphorous loosing.

Moreover, there is part of this liquid that cannot be recycling back to soft leaching for two main reasons: fulfil the global water balance and to bleed soluble impurities. The excess of liquid is sent to an effluent treatment stage that consists on a neutralization unit. The aim of this unit is to finally produce a purified waste water stream to be fed to sewage treatment plants and a solid by-product that can be used by the cement industry.

Hubert Halleux, Prayon, Belgium
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Senior Process Engineer

"Get More P": New Technology For Di-Calcium Phosphate Production From Low Grade Phosphate Sources

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In a context of decreasing quality of conventional phosphate raw material and demand for recovering and recycling of Phosphorus. Prayon Technologies has developed a new process route that upgrades such materials into valuable Di-Calcium Phosphate.

Following demographic expansion, the demand for phosphatic nutrient is continuously increasing. The quality of the conventional or secondary sources of phosphate is decreasing with lower P_2O_5 content and higher amount of impurities. It is difficult to process these "low-grade" materials in the conventional phosphoric acid production routes.

This process consists of 2 main steps:

1. an attack of the raw material by sulphuric acid in diluted condition to solubilize P_2O_5 followed by different steps to separate the impurities
2. a neutralisation of the filtrate produced at first step with a calcium source to produce a Di-Calcium phosphate. This DCP can be up-to animal feed grade.

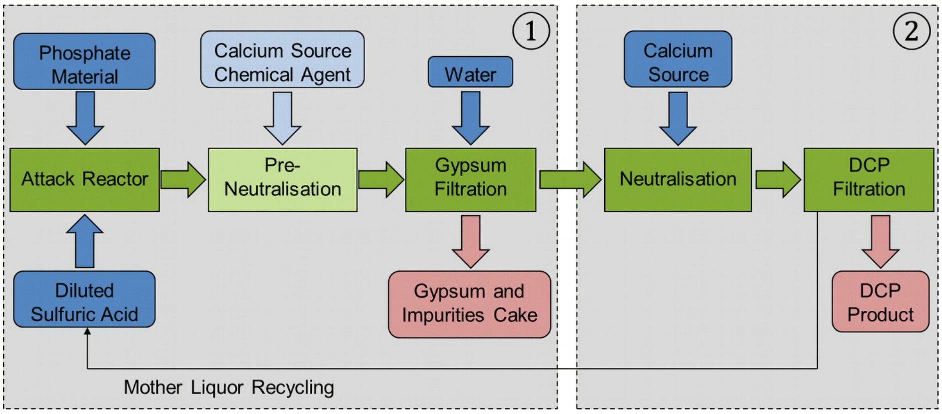

Figure 1: Simplified block diagram of "Get More P" process

Prayon has tested this process at lab and pilot scale on various phosphate source. These sources were mainly calcium phosphate and good results were obtained:

- P_2O_5 recovery up to higher than 90%
- Elimination of impurities (Fluorine, heavy metals, …)
- Production of good quality DCP

A few tests were realized on iron phosphate at lab scale with the following findings:

- P_2O_5 recovery lower than expected (around 60%
- Precipitation of iron phosphate during pre-neutralization reducing the global recovery of the process
- Higher consumption of calcium carbonate as there is no calcium in the raw material

This process needs complementary tests to assess the feasibility of processing iron phosphate-based sources.

References:

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Bengt Hansen, Kemira, Sweden

Mr Hansen works as an Application Manager in Kemira. He has 30 years of experience in the water, wastewater and sludge treatment field. Most of his career he has worked with application development as well as R&D and been involved in projects on five continents. He works with both drinking and waste water customers, mainly municipal. His core interest is energy optimization of wastewater treatment plants, phosphorus recycling and running ultra-distances.

How iron in sewage biosolids and recovered fertiliser products impact crop P availability

Bengt Hansen

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This short presentation shows results from some studies on phosphorous availability from sludge and iron phosphate. It is not enough to study the solubility of phosphorous, field and pot trials show that phosphorus is available even when solubility is low. Both municipal sludge, containing iron precipitated phosphorus, and iron phosphate can be used as phosphorous fertilizer.

Already 1932 *McGeorge and Breazeale* studied plant availability of phosphorus from aluminium and iron phosphates. They demonstrated that both aluminium and ferric phosphate showed an excellent assimilation of phosphate for wheat. A higher alkalinity reduced the uptake.

Around the millennium *Richards and Johnston* studied the availability of phosphorus in ferric phosphate amongst other recycled phosphate salts and compared it with no addition of phosphorus fertilizer at all and with mono calcium phosphate (MCP). The study was done in pot trials with ryegrass on 2 different soils that had different pH and phosphorous content. The grass was harvested 7 times during the 100days trial.

The results showed that a ferric phosphate could produce the same amount of dry matter grass as MCP when a slightly acidic soil was used (pH=6,6). The dry weight yield was about doubled compared with when no phosphorous fertilizer was added. When soil pH was 7,1 the MCP had a bit higher dry weight yield than ferric phosphate that had a double yield compared wit zero sample.

These trials also showed that the availability depends on time. After the first 3 harvests, the phosphorous uptake was higher for MCP but then ferric phosphate “catched up” and extracted more phosphate from the soil than MCP. After 5 harvests the same amount of phosphorous had been extracted from the soil. The soil chemistry developed equilibriums that ensures that the phosphorus can be taken up no matter of its origin in these trials.

O’Connor et al. conducted several studies on different organic fertilizers/biosolids in Florida. Their main target was to study erosion when different organic fertilizers were used. Among the organic fertilizers was manure, sludge from a biological phosphorous removal plant, sludge from WWTPs with chemical treatment, dried sludge etc. Triple superphosphate was used as a reference.

O'Connor et al. also studied the phosphorus availability. They analysed the phosphorous solubility with different extraction methods, phosphorus uptake and plant yield. When plotting the extracted phosphorus concentrations for the different fertilizers against the plant yield it is hard to see. When extracted with oxalate or citrate, there was no correlation at all but with water there was a poor correlation between yield and solubility, R^2 was 0,4.

Other conclusions from the study was that when Al and Fe content was high (mol Me:mol P >2) the yield dropped a few percent. But it was also clear that when the organic fertilizer contained iron or aluminium, the erosion was significantly reduced.

Hushållningssällskapet in South Sweden (*Per-Göran Andersson*) started a trial 1981 with 2 field trials with 2 different sludge. Both originates from WWTPs that mainly use iron salts for phosphorus removal. The trial is still on-going and the latest report describes the first 34 years of utilizing sludge. The same fields have been used all the time. 9 different combinations are tested, with or without sludge, with or without NPK and with low or high dose of sludge and the combination of these. The amount of N, P and K added varies but is never higher than the expected need for the crops.

34 years of trial shows that sludge increase the yield by 16% compared with if no fertilizer is added to the soil. When sludge and NPK was combined, the yield increased in average by 7% in average. This corresponds to an increased productivity value of 50-100 €/ha*year. There was no difference in crop uptake of cadmium or other metals. When sludge was used at high dosage, the metal content increased some during the first decade since sludge quality was poorer. It has stabilized after that.

Conclusions

Iron (and aluminium) precipitated phosphate is available for plants. It might not always be directly available but will be over time. When sludge, containing iron phosphate, is used in combination with NPK fertilizer the yield increase in average by 7% even if the same amount of n, p and K is added.

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I was trained in Environmental Science and Technology at TU Berlin with a specialization in Urban Water Management, Applied Soil Science and Environmental Analysis. After working in Soil Consultancy, I am currently a doctoral student working on the topic: 'Controlling of phosphorus fluxes in urban systems: Analogous processes in limnic sediments and sewage sludges'.

Iron-Phosphorus Coupling in Lake Sediments and Wastewater Treatment

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Controlling the key processes of iron (Fe) and phosphorus (P) coupling in lake sediments under anoxic conditions enables optimization of lake management measures preventing eutrophication by diminishing of P availability due to stimulating the P sink function of lake sediments. The key processes are P desorption and release into the water after reduction of Fe(III)[hydr]oxides, P immobilization under anoxic conditions by Fe(II) and the competition of P with other Fe(II) binding partners. Under the premises of an oxic water column and a minor amount of competing binding partners for Fe(II), redox stable Fe-P coupling can immobilize P in anoxic sediments.

In Fe rich lakes and sewage treatment plants (STPs) with application of Fe dosing, the Fe-P coupling changes due to the onset of Fe reducing conditions. The redox potential decreases in lakes from the water column towards the sediment and within the sediment, and in STPs from an aerated sludge system to the sludge digestion. In both cases, this is caused by the degradation of biogenic material. Due to these similar dynamics and processes, STPs can serve as „large scale laboratories" for lakes. The main goal is to optimize lake management measures to prevent eutrophication by P immobilization under anoxic conditions benefitting from Fe by controlling the key processes.

The Fe-P coupling, in lake sediments under anoxic conditions is controlled by three processes:

1. P release: Under oxic conditions Fe(III) has a low solubility and is precipitated as Fe(III)[hydr]oxides binding P well by adsorption in the water and at the sediment surface. When these Fe-P particles reach the anoxic sediment, Fe is reduced. Due to the higher solubility of Fe(II), it dissolves in the pore water together with the previously adsorbed P and both can diffuse towards the water column and deeper sediment. If meanwhile the water column has become anoxic, Fe(III) will not precipitate again and cannot adsorb P. In this case, P can enter the water column and become available for algal growth. However, if in the water column Fe oxidizing conditions persisted, Fe(III)[hydr]oxides can bind P again and the process, known as the Fe wheel, starts all over.

P immobilization: The simultaneous release of P and reduced Fe(II) can lead to an immobilization of P regarding anoxic conditions by mineral precipitation (Rothe et al., 2016). This process is exemplified by case studies of a naturally Fe rich lake and an Fe dosing STP. Sediment and sludge samples along the redox gradients of both systems were analyzed by a sequential extraction (Hupfer et al., 1995; Psenner et al., 1984). In both exemplary field studies, redox sensitive Fe bound P decreased with decreasing redox potential, whereas redox stable Fe bound P increased. Microscopy and XRD demonstrate that the redox stable P binding form was related to the Fe(II)-P mineral vivianite.

Competition with P binding: A case study of a lake amended by Fe addition illustrates that even an artificial Fe surplus did not necessarily initialize redox stable Fe(II)-P coupling. Instead of P, sulfur (S) coincided with Fe due to pyrite formation. S is not the only possible competitor with P for Fe(II), but it is the most prominent one in lake sediments. The competition with other Fe(II) binding partners reduces the capacity of Fe to immobilize P in lake sediments (Kleeberg et al., 2013) and, therefore, can increase P mobilization.

How can this knowledge help us to optimize lake management measures preventing eutrophication by diminishing P release and stimulating lake sediments as a P sink? An oxic water column prevents P mobilization to the water column and, by delivering the binding partners to the sediment, enables Fe(II)-P immobilization under anoxic conditions. Competitors binding Fe(II), like S, need to be included in the planning of management measures making use of Fe-P coupling under anoxic conditions. Under these premises, redox stable Fe-P coupling can immobilize P in anoxic sediments.

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Stefan Jansen, Deltares, The Netherlands

Stefan Jansen is senior researcher/advisor at Deltares, an independent Dutch knowledge institute for soil and water. His work focuses on biogeochemical processes in soil and water, including solutions for diffuse emissions of nutrients. He loves to design innovative, simple, but robust solutions and test them in the field together with farmers, water managers and other stakeholders.

Use of iron sand in agricultural drain systems to prevent P run-off

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Abstract summary

Iron oxide coated sand, a side product from drinking water production from groundwater, can be an excellent filter material which removes phosphorus to very low levels. By integration with the drainage system, efficient removal of P can be combined with minimal disturbance of the agricultural practice of the farmer. Field trials at several Dutch farms have shown the potential of various technical designs and the open questions that have to be answered before going to further application.

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Jansen, S., Gerritse, J., Stuurman, R., Chardon, W., Talens, R., 2016, Robust options to remove nitrate and phosphate from tile drainage. 2016 10th International Drainage Symposium Conference, 6-9 September 2016, Minneapolis, Minnesota. (doi:10.13031/IDS.20162514798)

Simon Kellmann, GEH Wasserchemie GmbH & Co. KG, Germany

Simon Kellmann holds a master's degree of Water Science from the University of Duisburg-Essen and he is specialized in water chemistry and microbiology. Since 2017, he works as a Business Development Manager at GEH Wasserchemie. His focus is on international business contacts and adaption of new water treatment technologies.

Regeneration of Phosphate-loaded Granular Ferric Hydroxide and P-recovery from Regeneration Solution

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Abstract

At a progressive rate, wastewater treatment plants need to be equipped with an additional phosphorus removal stage to achieve a good chemical status in the receiving natural water bodies. On the other hand, rapid growing demand of P-containing fertilizers calls for an effective recovery, as the world resources of phosphorous containing minerals are limited.

GEH® is an adsorbent, made up synthetically of pure granular ferric hydroxide, containing the mineral phases akageneite and ferrihydite. It was developed in the 1990s for arsenic removal in drinking water treatment and is successfully applied worldwide for this purpose [Driehaus et al. 1998].

Due to the chemical similarity, phosphate is also adsorbed selectively by granular ferric hydroxide. Therefore GEH® adsorption systems can be used effectively to counteract eutrophication in surface waters like lakes or ponds, but also in aquariums. Typical applications deal with relatively low P-concentrations (0.1-1.0 mg/L P) and the target is a reduction below detection limit. In municipal wastewater, phosphorous loads are typically higher, which leads to significantly higher loading rates of phosphate on the adsorbent. This generates the potential for P-recovery and processing for a commercial usage. The challenges in the wastewater application are the organic and solid content, which lead to high backwash frequencies or a necessary pre-treatment like a filtration step [Sperlich 2010].

The objective of our research was to investigate suitable techniques for regeneration of exhausted GEH® adsorbents and to recover the phosphorous from regeneration solutions. The adsorption experiments were conducted in laboratory column trials using model water, as well as in pilot filters with 15 kg GEH® using real municipal wastewaters. The phosphorous concentrations in the raw waters ranged from 10 to 20 mg/L P. The saturated GEH® material achieved a typical loading rate of 25 g P/kg GEH® (dry weight).

The regeneration of the loaded adsorbent was realized with an innovative three-step process, consisting of (1.) an acidic rinsing with hydrochloric acid at pH 2.5 to remove lime deposits, (2.) the

phosphate desorption step using 1 M sodium hydroxide solution (pH 14), and (3.) neutral rinsing of the adsorbent with HCl acidified tap water [Kunaschk et al. 2015]. One objective of the trials was minimizing the required volume of regeneration solution, while maximizing the regeneration efficiency. Depending on the applied process parameters (like contact time and volume of regenerate solution) a P-desorption in the range of 60 to 95 % was achieved. The remaining P-loading of the regenerated adsorbent ranged well below 1 g P/kg GEH® (dry weight), which makes it available for the next application cycle. However, the reduction of removal capacity after several regeneration cycles must be determined.

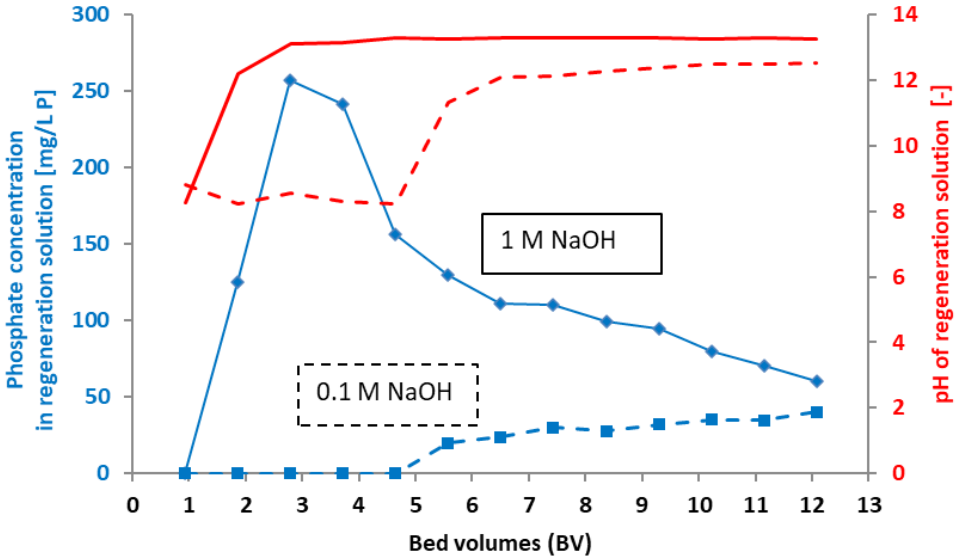

Fig 1: Phosphorous desorption from loaded GEH® using 0.1 M or 1 M NaOH solution in laboratory column tests

The highly alkaline regeneration solutions contain concentrations of up to 250 mg/L P and a notable organic content. The P-recovery from the solution takes place by a precipitation with lime water. As precipitation product, $Ca_3(PO_4)_2$ is formed, with a typical P-content of 20 % P_2O_5. The precipitate is filtered, washed, and dried and can be used as raw product for fertilizer production. An important measure for the practical application is the plant bioavailability (citrate solubility), which ranges 40-60 %. There is also no problematic accumulation of heavy metals happening. The remaining NaOH solution can be reused for desorption process again.

Acknowledgements:

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Links
https://www.geh-wasserchemie.com/en/applications/environment/

https://www.researchgate.net/lab/R-D-Lab-of-GEH-Wasserchemie-GmbH-Co-KG-Carsten-Bahr

Guy Kirk, Cranfield University, UK

Professor of Soil Systems at Cranfield with 30 years of experience in the biogeochemistry and modelling of soils and plant-soil interactions. Before joining Cranfield in 2003 he spent 13 years at the International Rice Research Institute, initially as Soil Chemist and later as Head of the Soil and Water Sciences Division, working with plant physiologists and breeders in developing rice with tolerance to soil constraints. Current/recent projects include 'Mechanisms and genetics of iron toxicity tolerance in African rice', 'Metal contamination of rice supplies in Asia', 'Rice germplasm for high grain Zn content and tolerance of Zn deficient soils'.

How iron in soil impacts root P uptake: Modelling rhizosphere effects

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Abstract summary

Some crop varieties are highly efficient in extracting phosphate from highly-weathered, iron-rich soils, which cover large parts of the humid tropics. However the mechanisms involved are not well understood and this limits progress in plant breeding and agronomy for such soils. This talk develops an earlier mathematical model of root-induced solubilisation of soil phosphate (Kirk, 1999) to account for measured rates of uptake by upland rice varieties with detailed information on root geometry and the role of soil microbes (Wissuwa et al., 2020).

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Siegfried Klose, EuPhoRe GmbH, Germany

After working in the international lime and fertilizer industry (including R&D and consulting), Siegfried Klose became self-employed in the area of trade and services for agriculture and environmental products in 1995. In addition to this business activity, he has been a member of the board of directors of the competence centre for fertilizers and the German phosphorus platform for many years. Since 2009 he has been significantly involved in the development of the patented EuPhoRe® process for the thermo-chemical treatment of sewage sludge with responsibility: raw material, product and quality management and market.

EuPhoRe® - recovery of phosphorus bound to iron in sludge

P-content bound to iron – long-term plant availability

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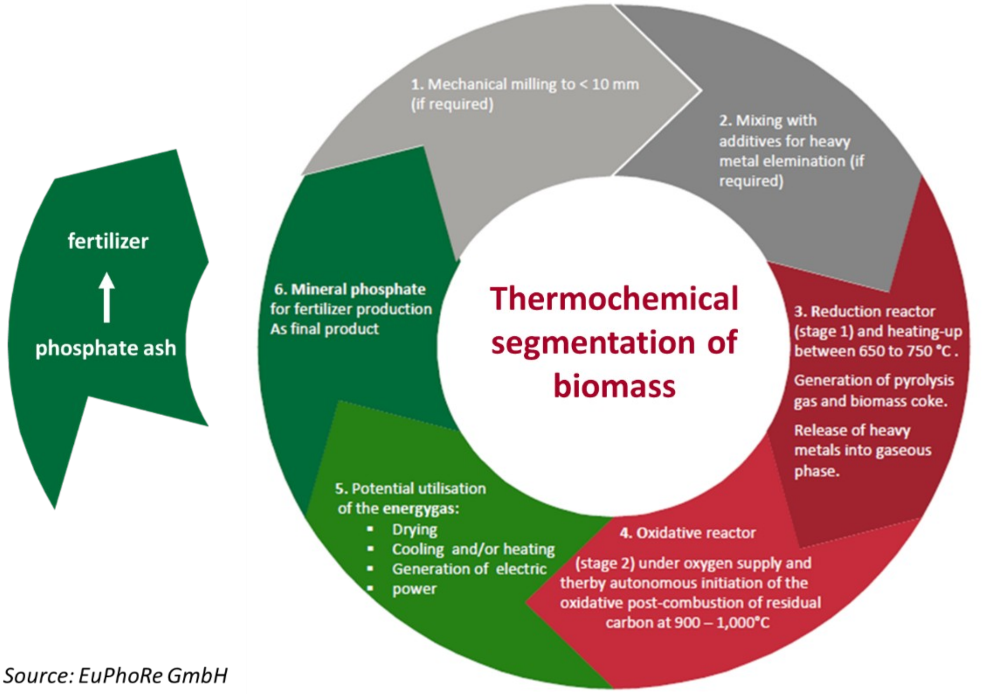
The thermo-chemical treatment of sewage sludge by the EuPhoRe® process generates an efficient long-term plant available P-recycplate. The P-content is bound to iron in the mineral type of Stanfieldit, further iron is bound in Magnetit while no Strengit (FePO4) is left.

1. Background EuPhoRe® processThe EuPhoRe® process is a thermo-chemical treatment of sewage sludge (fig. 1: process steps) to generate a fertilizer containing P that is readily available to plants.



Source: EuPhoRe GmbH

The resulting ash product contains low carbon while relevant heavy metal compounds and organic harmful substances are eliminated to a degree that all physical-chemical requirements of a fertilizer definitely are met at the end of the process. In addition, the ash contains nearly the entire phosphate load of the input material (> 98 %) and is eligible for use in agriculture after grinding and dust bond (e.g. via granulation).

Both an internationally funded pilot plant (please refer to video recording: Interreg, Phos4You)[1] and large-scale implementation in Switzerland and Germany are in the operating and implementation phase.

2. P-plant availability of P-recyclates – impact of iron

Current studies on P-plant-availability of P-recyclates (University of Gießen (D); 2020)[2] have confirmed the existing research results (HGoTech GmbH, Bonn (D); 2017)[3] that the EuPhoRe®-phosphates are already coming close to the TSP performance in P-uptake and even closer in the dry matter yield.

XRD analyzes showed that typical Fe precipitation phosphate Strengite ($FePO_4$) cannot be detected in the ashes generated by the EuPhoRe® process. Merrillite, Stanfieldite and Defectapatite are formed as phosphate phases in the given order. In the plant growing process, Merrillit is available to plants at short term, while the other two phases continuously release P causing a long-term plant availability. This had been as well documented in the soil investigations accompanying the experiment.

A small part of the Fe contained in the sewage sludge is found in the Stanfieldite, while the predominant Fe part is present in the Magnetite as FeO / Fe_2O_3. As the test results from HGoTech GmbH Bonn and University of Gießen confirm, Magnetite has no negative impact on P uptake, dry matter yield and consequently use efficiency of the fertilizer product.

It can be concluded that the overall evidence of the EuPhoRe®-process's application efficiency is provided.

3. References

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Leon Korving, Wetsus, The Netherlands

Leon Korving is scientific project manager at Wetsus. Wetsus is a European centre of excellence for sustainable water technology and develops breakthrough water technology together with more than 100 companies and 20 universities. Leon has a chemical engineering background with 20 years of experience in sewage sludge treatment and resource recovery from sludge, in particular phosphorus and coordinates the research at Wetsus on phosphorus recovery.

Philipp Wilfert, IPP, Germany

Dr. Philipp Wilfert did his bachelor and master theses related to biogeochemical processes in limnic and marine sediments respectively. The studies where supplemented with research stays in Namibia, Ireland and China. His PhD thesis phosphate release from iron phosphate containing sewage sludge"was made under the supervision of Professor Mark van Loosdrecht and Professor Geert-Jan Witkamp at TU Delft and Wetsus in Leeuwarden. Since two years Philipp works at the German engineering office IPP as a project manager in national and international projects related to urban wastewater treatment installations.

Vivianite formation and recovery from sewage sludge using magnetic separation or sulphide release

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Abstract summary

In this presentation we will present our findings on the interaction between phosphate, sulphide and iron in sewage sludge. We present evidence that vivianite is the main mineral precipitate and will also show how it interacts with sulphide. Furthermore, we show that the paramagnetic properties of vivianite can be used to recover it from sewage sludge using wet magnetic separators.

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Kees Langeveld, Fertilizers Europe, Belgium

Speaker is a chemical engineer working of 40+ years in the fertilizer industry mostly for ICL in various management functions.Representing here the producers representative Fertilizers Europe. During many years active in Circular Economy projects and realizing use of sewage sludge ashes, struvite and meat & bone meal ashes in the production units of ICL in Holland and Germany. Project manager of Project RecoPhos (P4 from sewage sludge ashes) and Project Tenova (food grade phosphoric acid from sewage sludge ashes). Active Board member in German Phosphate Platform DPP and Dutch Nutrient Platform as well as member of European ESPP

Iron Phosphates in the Fertilizer Industry

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Abstract summary

The European fertilizer industry has been active over the last decade to play a role in the Circular Economy movement. Also, for vivianite technologies this industry sees possibilities to be part of the value chain by using this product as P2O5 source and as Fe-source for deficient crops/soils or as fungicide in combination with either commodity or specialty formulations.

Changyong Lu, University of Copenhagen, Denmark

Changyong Lu, postdoc research at Department of Plant and Environmental Sciences in University of Copenhagen. PhD of material science 2017 - Universitat Autònoma de Barcelona. My current research is mainly about P recovery from wastewater. Two fields have particular focus: i) magnetic particles, such as magnetite anchored layered double hydroxide (LDH) composite, for P recovery, and ii) novel iron oxide nanomaterials for P removal, such as La doped single sheet iron oxide, Mn-Fe LDH.

Magnetic MgFe LDH composites for phosphate removal - is the LDH sufficiently stable?

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MgFe LDH could be dissolved under constant acid and neutral pH, even at alkaline pH the dissolution of MgFe LDH can also be observed under high P concentration. The sorption mechanism indicate the P removal was mainly via sorption on ferrihydrite at acid and neutral pH, sorption on LDH and formation of magnesium phosphate precipitates under alkaline pH. The instability of LDHs would hinder their practical application for wastewater treatment.

Abstract: Phosphate capture by magnetic sorbents is a promising technology for fast sorption of phosphate from large volumes of water. After exposure of the sorbents to waste water, the sorbents are captured magnetically and the sorbed phosphate can be extracted (and recycled) while the sorbent can be reused. Layered double hydroxides (LDHs) have been a popular sorbent as part of such magnetic particles due to the high sorption capacity of the LDH. However, there has been little attention to the stability of the LDH component.

We have synthesized magnetic sorbent particles consisting of a magnesium-iron(III) LDH linked to magnetite. Phosphate sorption isotherms showed high sorption capacities ranging between 3 mg P/g and 36.6 mg P/g at initial solution concentrations of 10 mg P/L, highest at low pH. However, the LDH component was highly unstable at pH below 9, with up to 50 % of Mg in LDH released to solution during 1 h. High P concentrations also stimulated LDH dissolution even at high pH. SEM, TEM, Mössbauer spectrosopy and XRD data demonstrate that various sorption reactions take place depending on pH and phosphate concentration. At acid pH, P is adsorbed to the amorphous poorly crystalized ferrihydrite phase produced when the LDH dissolves. At neutral pH range, the LDH dissolves to a less extent and the release of Mg^{2+} is partially suppressed. Hence, P is sorbed on the remaining Mg_3Fe LDH as well as the poorly crystalized ferrihydrite phase that has formed. Finally, at alkaline pH, Mg_3Fe LDH is the primary sorbent of P at low P concentration. At higher P concentration, the Mg_3Fe LDH phase is not stable and magnesium phosphates precipitate. Thus, P retention in the LDH system takes place via precipitation, and adsorption to Mg_3Fe LDH and the poorly crystalized ferrihydrite phase - depending on pH. This study illustrates the instability of the LDH phase

that strongly limits their application as recyclable sorbents. Future focus should be on reducing the solubility of the LDH component.

References:

Changyong Lu, Tae-Hyun Kim, Jesper Bendix, Mustapha Abdelmoula, Christian Ruby, Ulla Gro Nielsen, Hans Chr. Bruun Hansen. (2020) Stability of magnetic LDH composites used for phosphate recovery (Just accepted by Journal of Colloid & Interface Science, Article reference YJ-CIS26644)

Rouven Metz, University of Vienna, Austria

Rouven holds a Master in Environmental Chemistry, University Bayreuth, Germany after completing a Bachelor in Geoecology at the same university.

Biogeochemical mechanisms influencing the bioavailability of P and Fe from vivianite

Rouven Metz[1], Walter Schenkeveld[2], Naresh Kumar[1], Stephan Krämer[1]

1) University of Vienna
2) Utrecht University

The importance for vivianite ($Fe_2(PO_4)_3 \times 8H_2O$) as P burial in natural and artificial reducing environments as well as its use as a valuable resource is being increasingly recognized [1]. Vivianite has been found to form in costal marine sediments, in Fe supplemented eutrophic lakes, and in digested sludge of wastewater treatment plants [2, 3]. The formation of vivianite along with the removal of P from the aqueous phase, provides an opportunity to close the gap in the modern P cycle. Due to the chemical composition with a high P:Fe ratio, vivianite is proposed as a promising recycled and long-lasting P- as well as Fe- fertilizer. Few studies have already tested vivianite for Fe limited, calcareous oxic soils, with a positive response on plant growth [4-7]. However, the behavior of vivianite as a thermodynamically metastable mineral phase under oxic conditions in soils is still not very well understood. It is suggested that the rapid oxidation of vivianite leads to the destabilization of the mineral structure and the subsequent formation of amorphous Fe(III)-phosphate santabarbaraite with metavivianite as intermediate phase [8]. This transformation might enable plants to acquire P and Fe with relative ease from this poorly crystalline secondary mineral phase. However, the composition of the soil solution, specially the ratio of Fe to PO_4, Ca^{2+}, Si, and other ions can influence the formation and/or kinetics of resulting secondary mineral phases by possibly interfering the crystallization and thus the mobilization of P and Fe [9-11]. Additionally, under Fe deficiency, various organisms and plants are known to exude low molecular weight organic acids, reductants, and siderophores to increase Fe-(hydr)oxide dissolution and thus the bioavailability of Fe [12, 13]. It was also shown, that existing Fe^{2+} can enhance the ligand promoted dissolution *via* a ligand catalyzed reductive dissolution [14]. Recently, a synergistic effect between siderophores and reductants, especially pronounced in oxic calcareous soils was observed [15]. While this mechanism is based on a previous reduction step, the reaction with the Fe(II)-mineral is still unresolved. Further, the role of humic substances in promoting or inhibiting vivianite dissolution is also not well understood.

With our study, we aim to determine the rates and mechanisms of Fe and P mobilization from vivianite using controlled laboratory experiment with model soil systems. Data thus obtained will be used to derive mechanistic models on the vivianite transformation kinetics in various environmental conditions. The primary goal of this study is the parameterization of kinetic models for vivianite oxidative and ligand promoted dissolution including the influence of geochemical parameters such as pH, ligand type and O_2 partial pressure.

Lisbeth M. Ottosen, Department of Civil Engineering, Technical University of Denmark.

Lisbeth has two major research lines are use of secondary resources in construction materials (mainly concrete and bricks) and recovery of elemental resources such as phosphorous and heavy metals from particulate materials (ashes, soils, sludges and sediments). Lisbeth is MSc in civil engineering and received her PhD within environmental technology. She is author of more than 170 ISI indexed Journal papers.

Separation of phosphorus from sewage sludge ash by electrolysis

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This presentation is on electrodialytic separation of sewage sludge ash (SSA) into resources. Sewage sludge incineration is practiced extensively in many European countries. The produced SSAs have high concentrations of phosphorous, which is an essential to all life. Phosphate rock is listed as EU critical raw material based on two indicators: high supply risk and equally high economic importance. Currently most P-fertilizers originate from mining of sedimentary phosphate rock deposits, but this source is finite and non-renewable. A more sustainable alternative is mining from secondary resources such as SSA.

In order to recover P from SSA it is necessary to develop separation technologies. This work focus on one such technology, namely electrodialytic separation [1]. In this method, the SSA is suspended in water and an electric DC field is applied. The suspension of SSA is placed in the anode compartment, which is separated from the cathode compartment by a cation exchange membrane (CEM). During the application of current, the SSA suspension gradually acidify, desorbing both P and heavy metals. During the process a separation occurs simultaneously to the release in that the positively charged heavy metals are electromigrating over the CEM, whereas the negatively or uncharged P remains in the anolyte.

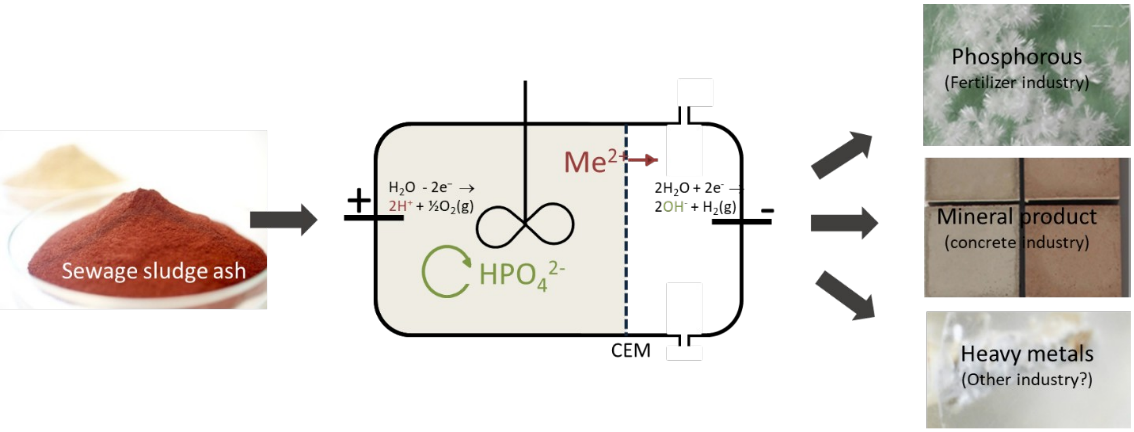

Figure 1: Electrodialysis separates sewage sludge into resources

During the electrodialytic separation of SSA two major resources are produced (figure 1): Phosphorous for the fertilizer industry and a mineral fraction for use as partly cement replacement in concrete. The ratio of heavy metals to phosphorous in the produced crystals is comparable to the very low end in phosphorous fertilizers at the market today [2]., which is a major environmental benefit in relation to the final fertilizer product. When using the mineral fraction of the SSA after recovery of phosphorous as partly cement replacement, the resulting concrete has an interesting aesthetical expression because of a warm red color, and the material properties were highly encouraging [3]. As cement production is a major anthropogenic CO_2 emitting process, replacement of cement in concrete with SSA could also give a major environmental benefit, in addition to resulting in a new interesting concrete type.

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Christoph Ponak, Montanuniversitaet Leoben, Austria

Dr. Christoph Ponak works as a researcher at the Chair of Thermal Processing Technology of the Montanuniversitaet Leoben, Austria. He studied Industrial Environmental Protection and Process Technology and his doctoral thesis focused on high-temperature recycling processes involving the reaction behaviour of elemental phosphorus. He is currently working on the development and improvement of processes for sewage sludge ash, steelmaking slag and Li-ion battery recycling. He has authored and co-authored more than 20 publications and conducted an outgoing research stay at The University of Tokyo. His teaching focuses on industrial environmental protection, high temperature process engineering as well as systemic sustainability.

Removal of gaseous phosphorus during the carbo-thermal reduction of sewage sludge ashes

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Abstract summary

The RecoPhos process and pilot plant (constructed and operated at the Chair of Thermal Processing Technology at a scale of 5-10 kgh^{-1}) enable the production of elemental phosphorus by full carbo-thermal reduction of sewage sludge ash. Its advantages include a flexible energy input (in order to treat cold ash as well as hot ash from a prior incineration step), complete independence from a variation of input material composition (e.g. varying iron and aluminium contents depending on precipitation salt use) and the possibility to simultaneously recover iron and other valuable metals. Currently, the engineering of a 50 kgh^{-1} reactor is carried out (construction of a pilot plant in 2021 in Leoben, Austria) and accompanied by the creation of a simulation model to design a semi-industrial demo plant with a scale of 350-500 kgh^{-1} (2022/23).

Abstract

The RecoPhos process aims at the production of gaseous, elemental phosphorus. While it is theoretically possible to produce white phosphorus, dual use issues have led to the construction of a pilot plant that post-combusts the phosphorus gas and applies a gas scrubber in order to produce phosphoric acid. Depending on the objectives of the operator, gas cleaning can be installed so that high quality acid can be withdrawn from the process at any desired concentration or acid that still contains volatile compounds from the sewage sludge ash can be sold to cleaning facilities.

The core component of the process is the so-called InduRed reactor. It is shown in Figure 1.

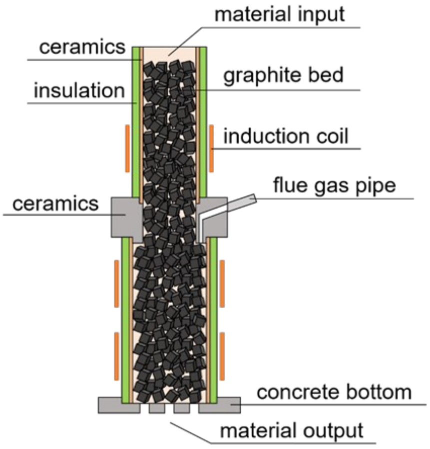

Figure 1: InduRed reactor

The reactor principle is based on an inductively heated bed of graphite pieces. It has the following distinct properties and advantages:

- operates with a (high) temperature profile that is evenly distributed across the whole reactor
- provides a large reaction surface
- provides short mass transport distances
- maintains a low pressure atmosphere
- is divided into several temperature zones so that pre- and post-processing steps can be optimized
- provides a possibility for gas removal close to the gas production

These properties are currently under investigation in terms of their suitability for lithium-ion battery treatment. Numerous synergy potentials (e.g. carbon from anodes as a reducing agent, iron and phosphorus separation if the lithium-iron phosphate type amount increases) can be detected.

Since the ash composition is dependent on the use of precipitation salts (iron- or aluminium-based) in waste water treatment plants, the composition – especially regarding the phosphate composition – might vary greatly. The presented reactor tackles this challenge by providing the possibility of flexible energy input in different zones by induction.

The induction system also enables flexibility when it comes to the origin of the treated ash. Cold ash needs more energy input than hot ash that is provided by a prior incineration step. Both can be processed and potentially even be adapted to energy prices.

Phosphoric acid is the main driver for financial feasibility, which has been analysed in great detail and which shows positive results (though with a dependence on gate fees). However, the oxidic compounds of the ash can be adjusted by using additives (mainly CaO and Al_2O_3) in order to produce a slag that is a suitable substitute for clinker in cement plants. This basic feasibility has already been confirmed by experiments and respective analyses. The metal phase can potentially be used for precipitation salt production or as an alloying additive.

The post-combustion of the CO- and P-rich product gas provides a possibility for heat utilisation as does the potential dry slag granulation of the product slag.

Since the process is highly complex, it has taken some time to be ready to seriously consider a scale-up process that has recently been kicked off. A simulation model combining heat input, multi-phase reaction kinetics as well as magneto-hydrodynamics is being set up in order to facilitate the step from 50 to 500 kgh^{-1}.

As of now, the following aspects have been tested and verified in multiple experiments:

- reduction of > 99 % of phosphorus compounds
- removal of > 75 % of the phosphorus as an elemental gas
- reduction of > 99 % of the iron compounds
- production of an iron- and phosphorus-free slag
- feasibility of dry slag granulation of the product slag
- good hardening behaviour by use of respective additives (clinker substitute possibility)
- economic feasibility

Ruben Sakrabani, Cranfield University, United Kingdom

Dr Sakrabani has more than 15 years of experience in determining nutrient dynamics in soils associated with application of organic amendments such as compost, manure, slurry, sewage sludge, biochar and digestates. His work explores the use of new approaches where prediction of nutrient release from organic amendments can be exploited in order to maximise resource efficiency and promote sustainable intensification of agriculture. He has a good track record being involved in projects on biosolids application to land in relation to its use as alternative phosphorus fertiliser. His recent work funded by Natural Environment Research Council (NERC) has resulted in the Phosphate Acceptance Map. He has published in over 60 peer reviewed journals and is currently the Associate Editor of Soil Use and Management. He is also member of the NERC Advisory Network and NERC Peer Review College.

Role of P analytical methods and their implications for evaluating P availability in crops

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Abstract summary

- Analytical methods for P need to be fit for purpose – suitability to calcareous or acidic soils
- Mismatch of analytical methods with soil can over or under estimate P bioavailability in soils
- The existing P analytical methods each have to be interpreted in terms of its use to assess bioavailability within a set boundary condition

Phosphorus (P) is a key macronutrient needed by crops, and its role is irreplaceable by other elements. This makes managing P a challenge as its application to soil has to be precise to meet crop demands. An additional challenge is that the major deposits of mined P are located only in certain parts of the world, which makes it essential that alternative sustainable sources of P are also considered in agricultural production. When considering either mined or sustainable sources of P, a key aspect is determining its content to ensure that an accurate supply can be applied to soil to support crop management. The current understanding in terms of plant availability of P in soil is that it exists in various pools; availability of P in the recalcitrant pools is very low, whilst the more readily available pools show greater accessibility by plant roots. Soil pH is a key driver that contributes towards the availability equilibrium, which regulates the dynamics of P pools in addition to temperature and texture (presence of clay). According to the pool concept, the immediately available pool of P is the one which roots can gain access to and is the smallest fraction. This poses a challenge as an accurate method to determine the immediately available pool is the key to ensure the administration of recommended quantities of P fertiliser.

The current approach to determining immediately available P is through chemical extractions, where a sample of soil is extracted using bespoke chemicals. Such extractions represent the portion of P in the soil that is available to plants roots. There are many different extraction procedures in place, which often corresponds to pH of the soil. P in acidic soils are usually determined using Bray 1, Mehlich-3 or Morgan whilst alkaline soils utilise either Colwell or Olsen. The various extraction methods cater for the different conditions and have demonstrated their suitability to meet the local context where experiments had been carried out. In addition to these extraction methods, there are also techniques which act as sinks for P analysis. These techniques include the use of Diffusive Gradient in Thin Films (DGT) and anion exchange resin beads as sinks designed to act as passive samplers as they do not involve active extraction using bespoke chemicals. Such sinks include a ferrihydride gel in DGT or bespoke resins that attracts P across a concentration gradient in soil, and adsorbs P into the sink which is then extracted using conventional method of analysis. More recent methods of determining P include non-wet chemistry technique using mid (MIR) and near infra-red (NIR) technology where a source of radiation is directed to soil samples to activate specific chemical bonds, which are then reflected and used to quantify the presence of a particular analyte (P). The MIR and NIR techniques are quick and can be carried out in-field conditions compared to the conventional wet chemistry methods involving extraction. However the MIR and NIR needs a spectral library to be able to calibrate the spectrum and interpret the data.

The choices of these various forms of P analysis are necessary to cater for the different needs of a particular project. Each method has its own strengths and weaknesses; it can be more valuable to use an extraction method in conjunction with a passive sampler in order to be able to predict P availability. There is little scope to harmonise the extraction methods but a robust soil sampling method that best captures the heterogeneity of the landscape to ensure representative sample is collected is key when using in conjunction with the most suitable and available method. This review demonstrates the high complexity of soils with multiple dependencies and interactions of different parameters. Hence, it is recommended that any statement assessing the plant availability of different P-compounds should include information about the applied method and the boundary conditions in the particular case.

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Caroline P. Slomp, Utrecht University, the Netherlands

Caroline Slomp is a Marine Biogeochemist. Her research focuses on the cycling of elements that are important to life in marine environments, such as carbon, phosphorus and iron. Her research involves seagoing field work, laboratory analyses and modeling. The central theme is the assessment of the response of marine environments to perturbations. She has ongoing projects on, for example, ocean deoxygenation, methane dynamics and trace metal cycling. Field work areas include the Baltic Sea, Black Sea and various coastal regions in the Netherlands, Sweden and USA. She also studies geological deposits to understand oceanic anoxia in Earth's past.

Phosphorus dynamics in coastal marine sediments: new insights in the role of Fe-P phases

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Abstract summary

In this presentation, I show that the Fe(II)-P mineral vivianite can be a quantitatively important sink for phosphorus in coastal marine sediments. I also discuss methods to determine vivianite in sediments and the conditions of formation.

Abstract

Phosphorus (P) is a key nutrient for phytoplankton in the marine environment. Increased availability of P can contribute to elevated productivity and the development of low oxygen zones in coastal systems. Phosphorus associated with Fe-oxides is traditionally thought to play a key role as both a temporary and permanent sink for P in such areas. Here, I summarize findings for sediments from the Baltic Sea demonstrating that Fe(II)-phosphates should also be considered as a potential temporary and permanent sink for P in coastal marine environments.

First, I show how the operational SEDEX extraction procedure for phosphorus in sediments can be complemented with micro-XRF, XANES, SEM and XRD analyses to identify and quantify Fe(II)-P phases in coastal sediments (Egger et al., 2015). Second, I discuss various mechanisms that contribute to formation of Fe(II)-P phases in sediments. In the Bothnian Sea, for example, variations in organic matter and iron input are suggested to act as a key driver of Fe(II)-P formation (Slomp et al., 2013; Egger et al., 2015; Rooze et al., 2016; Lenstra et al., 2018). In this setting, vivianite is formed below a shallow sulfate methane transition zone (SMTZ) in the upper 10-20 cm of the sediment. The Fe(II)-P acts as a major burial sink for P and its burial may provide a negative feedback on further eutrophication of the Bothnian Sea region. Using reactive transport modeling, we show that a low bottom water salinity is especially conducive to vivianite formation in coastal sediments (Lenstra et al., 2018). Results of sulfidization experiments highlight the sensitivity of vivianite to exposure to sulphide. Hence, vivianite formed in the sediment is only preserved if it is not exposed to sulphide during long-term burial (Dijkstra et al., 2018).

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Hui Xu, Ghent University, Belgium

A post-doc researcher working on project of 'Upscaling and prototype development of phosphorus filters for agricultural drainage waters'. We aim to develop a filtering system to remove different forms of phosphorus from agricultural drainage systems.

Reducing phosphorus (P) losses from drained agricultural fields with iron coated sand (ICS) filters

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Abstract summary

In Belgium, agricultural diffuse phosphorus (P) loss is a major cause of eutrophication problems in surface water and our research group of Soil Fertility and Nutrient Management developed small-scale prototype filters (see the figure) for removal of P from drainage water. Performance of these filters was evaluated in the different fields and consequently, more than 80% of dissolved reactive P and total P were removed over seasons. These filters showed to have the potential to improve the water quality significantly.

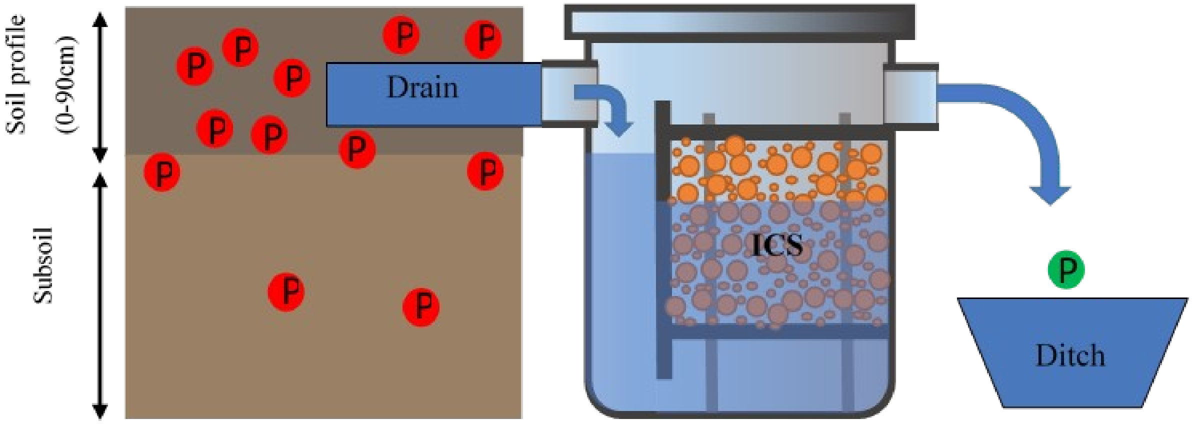

Figure: Schematic representation of prototype filter in the field

Reference:

Vandermoere et al. (2018). Reducing phosphorus (P) losses from drained agricultural fields with iron coated sand (- glauconite) filters. Water research 141, 329–339. doi:10.1016/j.watres.2018.05.022

Links

A video about our project: https://www.youtube.com/watch?v=zhfOgH1AnME&t=12s
Information brochure of our filter: https://northsearegion.eu/media/13649/icsf-fact-sheet-200513.pdf